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Summary of Rocky Flats Plant Waste Buried in the Subsurface Disposal Area

Edward Vejvoda

**Idaho
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April 2005

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ABSTRACT

This report addresses characteristics, nomenclature, and generating processes associated with radioactive and mixed waste shipped from Rocky Flats Plant to Idaho National Laboratory from 1954 to 1989. Until 1970, the waste was buried in the Subsurface Disposal Area, located in the Radioactive Waste Management Complex at Idaho National Laboratory.

The report reviews the types of waste, packaging, assay methods, transportation arrangements, waste identification labeling, and communications involved between Rocky Flats Plant and organizations at Idaho National Laboratory. An extensive set of appendixes is provided consisting mainly of Rocky Flats Plant documentation pertinent to the topics being presented.

FOREWORD

The author has 52 years of experience in the areas of plutonium and uranium operations gained primarily at Rocky Flats Plant. He was an active employee at Rocky Flats Plant from 1952 to 1987 and a consultant for 17 years. Mr. Vejvoda started employment at Rocky Flats Plant in June 1952 and retired in 1987. He returned as a consultant to Rockwell, EG&G, and Kaiser-Hill contractors until 2003. He holds bachelor's and master's degrees with a major in chemistry and a minor in physics.

His work experiences and positions include an initial assignment in 1952 to the spectroscopy laboratory for highly enriched uranium in Building 881, followed by advancement to the Plutonium Analytical Methods Development Group in 1956. From 1965 to 1974, he was the manager of the Chemical Technology Group, which carried out special projects and recovered plutonium and other actinides from scrap and residues not acceptable for the regular plutonium recovery stream. The Chemical Technology Group generated the majority of waste contaminated with Np-237, Cm-244, U-233, and other actinides used in the radiodiagnostic tracer program. The Plating Laboratory—located in Building 444 and part of the Chemical Technology Group—and the Special Recovery Group were also under his supervision.

In 1974, he became Director of Chemical Operations, which included plutonium recovery operations (Building 771) and the molten salt extraction process located in Building 776. Later, he was assigned the responsibility of director of all plutonium operations, which included pit manufacturing and assembly, plutonium recovery, pyrochemistry processing, and waste management. The waste management assignment consisted of solid waste processing, liquid waste treatment, and waste packaging and shipping.

During his operational tenure, he experienced and worked with the gradually increasing stringency of requirements of waste management practices. His experience with plutonium recovery and special recovery operations provided insight into the generation of both solid and liquid waste treatment and waste shipping requirements. Plutonium recovery activities were the major processes contributing to waste shipped to Idaho National Laboratory.

He has served as consultant to Idaho National Laboratory personnel in the areas of waste identification, waste shipping records, and Rocky Flats Plant operations and facilities. Over a 52-year period, he has experienced the Rocky Flats Plant startup, mission assignments, and decommissioning and cleanup phase.

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The author wishes to acknowledge the assistance of former Rocky Flats Plant employees. K. W. Calkins, a 30-plus-year former employee, provided technical input on economic discard limits and chemical engineering plutonium upgrade projects. R. W. Woodard, another long-time Rocky Flats Plant former employee, provided technical information related to highly enriched uranium processing and data about high-efficiency particulate air filters. S. L. Digiallonardo helped with his operating experience in Buildings 444 and 776. Additional waste information related to plutonium residues and processing of high-efficiency particulate air filters was provided by Jack Weaver. J. K. Frettholder, a consultant engineer with the U.S. Department of Energy Rocky Flats Plant Operations Office, contributed data and explanations about the history of high-efficiency particulate air filters at Rocky Flats Plant.

Personnel at Idaho National Laboratory, such as Rod Thomas, Bruce Becker, and Danny Anderson, were helpful through their questions and topics of interest. Many thanks to Vivian Schultz and staff, who produced this report and assisted with its organization, and to Ed May and Tasha Taylor for editing. This report was sponsored and encouraged by Jean Holdren, who also had the patience to see it through to completion.

CONTENTS

ABSTRACT.....	iii
FOREWORD	v
ACKNOWLEDGMENTS	vii
ACRONYMS.....	xix
1. INTRODUCTION.....	1
2. BACKGROUND.....	3
2.1 Rocky Flats Plant Facilities (1953–1969)	3
2.2 Additions to Rocky Flats Plant Facilities (1970–1989)	5
2.3 Beryllium Processing	6
3. WASTE OPERATIONS AND ORGANIZATION	7
3.1 Waste Disposal Unit	7
3.2 Waste Operations	7
4. WASTE CHARACTERIZATION.....	9
4.1 Liquid Waste Treatment.....	9
4.1.1 First- and Second-Stage Sludge	10
4.1.2 Off-Specification Waste Solutions	11
4.1.3 Evaporator Salts	11
4.1.4 Contaminated Drums	11
4.1.5 Treatment of Organic Liquid Waste.....	12
4.2 Solid Waste Treatment	12
4.2.1 Line-Generated Waste	13
4.2.2 Sludge Waste	13
4.2.3 Filter Waste.....	13
4.2.4 Maintenance Operational Waste	13
4.2.5 Non-Line-Generated Waste	14
4.3 Isotopic Levels in Waste	14
4.4 Waste Constituents	15
4.4.1 Combustible Waste—Type I.....	15
4.4.2 Filter Paper—Type II	16
4.4.3 Chemical Warfare Service Filters—Type III	17

4.4.4	Sludge—Type IV	17
4.4.5	Noncombustible Waste—Type V	17
4.4.6	Waste from Buildings 444, 447, and 883	17
4.4.7	Building 881 Waste	21
4.4.8	Buildings 122 and 123 Waste	22
4.4.9	Building 991 Waste	22
4.4.10	Building 886 Waste	23
4.4.11	Sewage Treatment Plant—Building 995	23
4.4.12	Building 331 Waste	23
4.4.13	Building 865 Waste	23
4.4.14	Plutonium Waste-Generating Facilities	23
4.4.15	Offsite Waste Sources	24
4.4.16	Waste Quality Program	26
5.	DEVELOPMENT OF WASTE CONTAINER STANDARDIZATION AND IDENTIFICATION	27
5.1	Generator Identification	27
5.1.1	Post-1970 Identification of Containers	30
5.2	Waste Containers	30
5.3	Waste Packaging	31
5.3.1	Non-Line-Generated Waste	31
5.3.2	Line-Generated Waste	31
5.3.3	Crated Waste	31
5.3.4	Series 774 Sludge Waste	32
5.3.5	Series 743 Grease Sludge	32
5.3.6	Neutralized Hydrochloric Acid Solutions	32
5.3.7	Post-1970 Packing	32
5.4	Packaging and Container Codes	32
6.	WASTE ASSAY	34
6.1	Liquid Waste Assay	34
6.2	Raschig Ring Tanks	35
6.3	Solid Waste Assay	36
6.4	Production Nondestructive Assay Systems	36
6.5	Plutonium Waste Discards	36
6.6	Economic Discard Limits	38
6.6.1	Background	38
6.6.2	Establishing Economic Discard Limits	38

6.6.3	Calculating Economic Discard Limits.....	39
7.	WASTE TRANSPORTATION	41
7.1	Trailers	41
7.2	Atomic Materials Rail Transfer Railcars.....	41
7.3	Piggy-Back Trailers	41
7.4	Shipping Forms.....	42
8.	NUCLEAR CRITICALITY SAFETY	48
8.1	Storage Criticality Limits	48
8.2	Transportation Criticality Limits	48
9.	PROCESS AND PLANT CHANGES.....	50
9.1	Housekeeping Waste.....	50
9.2	Maintenance Waste.....	50
9.3	Process Waste.....	50
9.3.1	Foundry and Fabrication Waste	50
9.3.2	Component Assembly and Return Disassembly	50
9.3.3	Production Support Activities	51
9.3.4	Chemical Recovery and Metal Recycle.....	51
9.3.5	Waste Treatment Waste.....	51
9.4	Major Contamination Incidents	52
9.5	Mission Changes.....	52
10.	SPECIAL-ORDER WORK	53
10.1	Radionuclide Tracer Program.....	53
10.2	Neptunium Program.....	53
10.2.1	Neptunium Acquisition.....	53
10.2.2	Neptunium Inventory.....	53
10.2.3	Neptunium Material Balance Area Accounts	54
10.2.4	Plutonium-Neptunium Alloy Preparation	55
10.2.5	Scrap and Residue Generation	56
10.2.6	Waste Generation	56
10.2.7	Summary.....	57

10.3	Uranium-233 Program	57
10.3.1	Scrap and Residues.....	57
10.3.2	Waste Generation.....	57
10.3.3	Processing Facilities	58
10.3.4	Summary.....	58
10.4	Americium Program.....	58
10.4.1	Americium Feed Sources.....	58
10.4.2	Americium Recovery Processes.....	59
10.4.3	Waste Streams.....	59
10.5	Thorium Program.....	59
10.6	Curium-244 Program	59
10.7	Other Special-Order Work	59
11.	CLASSIFIED WASTE.....	60
11.1	Shipment of Contaminated Classified Material.....	60
12.	SPECIAL TOPICS	62
12.1	Graphite Waste	62
12.1.1	Plutonium Graphite Molds.....	62
12.1.2	Plutonium Discard Limits	66
12.2	Roaster Oxide	66
12.2.1	Roaster Oxide Description.....	66
12.2.2	Chip Roaster	66
12.2.3	Casting Skull.....	67
12.2.4	House Vacuum System.....	67
12.2.5	Sintered Metal Filter Collections	67
12.2.6	Water-Quenched Tanks	67
12.2.7	Furnace Box Stubs	67
12.2.8	Packaging Roaster Oxide.....	67
12.2.9	Roaster Oxide Pyrophoricity.....	67
12.2.10	Roaster Oxide Shipments.....	68
12.3	Ion Exchange Resins.....	69
12.4	Decommissioning Building 881	69
12.5	Discard Mud.....	69
12.6	Plutonium-Contaminated Oralloy Waste	69
12.7	Oralloy-Contaminated Equipment	70

12.8	1969 Fire Waste.....	70
12.9	903 Pad Disposal	70
12.10	Asbestos and Miscellaneous Waste Items.....	71
12.11	High-Efficiency Particulate Air Filters	72
12.11.1	Background.....	72
12.11.2	Plutonium Ventilation Systems.....	73
12.11.3	Before 1964.....	73
12.11.4	Uranium Processing Building	75
12.11.5	High-Efficiency Particulate Air Filter Plutonium Measurements	76
12.12	High-Efficiency Particulate Air Filter Management.....	77
12.12.1	Plutonium Exhaust Ventilation System	77
12.12.2	Filter Background.....	77
12.12.3	Filter Generation Rates	78
12.12.4	Filter Configuration.....	79
12.12.5	Filter Media.....	79
12.12.6	Filter Discard Limits.....	80
12.12.7	Plutonium Filter Content	81
12.12.8	Plutonium Recovery from Spent Filters	81
12.12.9	Filter Processing.....	81
12.12.10	Waste Filter Packaging.....	82
12.13	Waste Drums Returned to Rocky Flats Plant	82
12.14	Mound Disposal.....	83
12.15	Reactor Grade Plutonium	83
12.16	Cyanide Waste.....	83
12.17	Glove-Box Gloves	83
12.18	Liquid Organic Waste	83
12.19	Sludge	85
12.19.1	Organic-Based Sludge	85
12.19.2	Acidic-Based Sludge	86
12.19.3	Caustic-Based Sludge.....	86
12.19.4	Organic-Aqueous-Based Sludge	86
12.20	Mercury Waste	87
12.21	Excess Chemical Compounds	87
12.22	Polychlorinated Biphenyls	87

12.23	Complexing Agents	87
12.24	Analytical Methods.....	88
13.	TIMELINE AND CORRESPONDENCE—WASTE MANAGEMENT.....	89
13.1	Timeline	89
13.2	Correspondence from Edward Vejvoda to Operable Unit 7-13/14 Staff	93
14.	REFERENCES.....	94
	Appendix A—Letter from G. V. Beard to John Epp.....	A-1
	Appendix B—Letters from Colorado School of Mines.....	B-1
	Appendix C—Packaging Certification by Atomic Energy Commission—ALO Contractor (August 21, 1967).....	C-1
	Appendix D—List of NDA Reports Available	D-1
	Appendix E—Handbook of the Rocky Flats Plant Production Non-Destructive Assay Systems (compiled by Bill Ulbricht).....	E-1
	Appendix F—Handbook of the Rocky Flats Plant Production Nondestructive Assay Systems (1987)....	F-1
	Appendix G—MUF Study of the Idaho Burial Field and Rocky Flats Drum Storage Area.....	G-1
	Appendix H—Plutonium Waste Discards CY67	H-1
	Appendix I—Sorting of Radioactive Waste at Rocky Flats	I-1
	Appendix J—Outside Storage for Drums of Building 71 Washables	J-1
	Appendix K—Storage of 55-Gallon Drums in Buildings 71, 76, and 34.....	K-1
	Appendix L—Letter to R. F. Rogers from J. D. McCarthy	L-1
	Appendix M—Nuclear Materials Safety Limits for Building 664.....	M-1
	Appendix N—Special Permit No. 5948	N-1
	Appendix O—Letter to V. C. Vespe from E. H. Lee	O-1
	Appendix P—Production Scale Americium Recovery at Rocky Flats 1953–1980.....	P-1
	Appendix Q—Courier Receipt.....	Q-1
	Appendix R—Waste Shipment Data.....	R-1
	Appendix S—Load Lists	S-1

Appendix T—Letter to J. S. Corbett	T-1
Appendix U—Graphite Specifications	U-1
Appendix V—Drum Counter Verification Studies (CRDL 940232-101C).....	V-1
Appendix W—Drum Counter Verification Studies (CRDL 940232-101D).....	W-1
Appendix X—Letter to Hazardous Materials Regulation Board, Department of Transportation, from William F. Romine.....	X-1
Appendix Y—Packaging Certification by Atomic Energy Commission—ALO Contractor (Rev. 3-7-69).....	Y-1
Appendix Z—Building 44 Discarded Waste Report— Sources of Roaster Oxide	Z-1
Appendix AA—Letter to R. D. Gaskins from William F. Romine.....	AA-1
Appendix BB—Non-Salvable Contaminated Waste for Disposal, “B” Plant (July 1964–September 1965)	BB-1
Appendix CC—Non-Salvable Contaminated Waste for Disposal, “B” Plant (February)	CC-1
Appendix DD—Building 883 Crate Number 8716215	DD-1
Appendix EE—Crated Wastes	EE-1
Appendix FF—Measuring Plutonium on Equipment and Machine Tools Removed from the Fire Area	FF-1
Appendix GG—Ventilation Filter Reports Available	GG-1
Appendix HH—Extension of Useful Life of Exhaust Air Filters after Ammonium Nitrate Loading	HH-1
Appendix II—Preliminary Design Criteria for CWS Filter Processing	II-1
Appendix JJ—Analysis of Waste Drums No. 771-7959 and 771-7961 Returned from the National Reactor Test Site	JJ-1
Appendix KK—Letter from E. S. Ryan to G. E. White	KK-1
Appendix LL—Investigation CCl ₄ Carbon Tetrachloride by R. W. Hawes.....	LL-1
Appendix MM—A Control Design for Plutonium Counting Systems.....	MM-1
Appendix NN—A Plutonium Waste Counter.....	NN-1

FIGURES

1.	Form ID-110A, “Solid Radioactive Waste Disposal Order and Shipment Data”	43
2.	Form ID-135, “Radioactive Waste Form”	44
3.	Handwritten Rocky Flats Plant waste disposal load list	45
4.	Page 1 of computerized Form ID-135, “Radioactive Waste Form”	46
5.	Page 2 of the computerized load list.....	47
6.	Four general configurations for graphite molds	63
7.	Plutonium ventilation system.....	78

TABLES

1.	Rocky Flats Plant production plants 1953–1970.....	3
2.	Constituents and weights for a 55-gallon drum	12
3.	Rocky Flats Plant plutonium isotopic levels in waste (stream averages—plutonium wt%).....	14
4.	Typical isotopic concentrations in waste	15
5.	Personal protection garments used in working with radioactive and hazardous materials.....	16
6.	Waste codes for Buildings 444 and 447	18
7.	Offsite shipments received by Rocky Flats Plant and shipped to Idaho National Laboratory.....	24
8.	Identification of waste container generator pre-1970	27
9.	Post-1970 transuranic and nontransuranic waste generators	29
10.	Bureau of Explosives codes on the load lists	33
11.	Liquid waste solutions received for treatment December 1961	35
12.	Suspect drums shipped to Idaho National Laboratory.....	37
13.	Suspect drum comparison.....	37
14.	Economic discard limits—Fiscal Year 1969.....	40
15.	Average plutonium values for drums shipped	49
16.	Neptunium-237 fiscal year-end inventories	54

17.	Neptunium material balance area accounts.....	54
18.	Classified waste shipment sites.....	60
19.	Summary of depleted uranium waste shipments to Idaho National Laboratory based on reports by the Waste Disposal Coordination Group.....	68
20.	903 Pad drum processing chronology	71
21.	Disposal summary of plutonium-contaminated materials, 903 Pad storage area.....	71
22.	Waste shipments to Idaho National Laboratory from Building 771.....	74
23.	Waste shipments to Idaho National Laboratory from Building 776.....	75
24.	Plutonium discard limits for filter media in 1992	76
25.	Summary of filters shipped to Idaho National Laboratory taken from reports prepared by the Waste Disposal Coordination Group	79
26.	Filter economic discard limits for 1968.....	80
27.	Organic waste.....	85

ACRONYMS

AEC	U.S. Atomic Energy Commission
CCl ₄	carbon tetrachloride
CWS	Chemical Warfare Service
DOE	U.S. Department of Energy
DOT	U.S. Department of Transportation
DU	depleted uranium
HEPA	high-efficiency particulate air (filter)
HEU	highly enriched uranium
INL	Idaho National Laboratory
MSE	molten salt extraction
NDA	nondestructive assay
NRTS	National Reactor Testing Station
OASIS	Organic Accelerated Solidification and Immobilization System
ORNL	Oak Ridge National Laboratory
PCE	tetrachloroethylene
PVC	polyvinyl chloride
R&D	research and development
RCRA	Resource Conservation and Recovery Act
RFP	Rocky Flats Plant
SNM	special nuclear material
TCE	trichloroethylene
TRU	transuranic

Summary of Rocky Flats Plant Waste Buried in the Subsurface Disposal Area

1. INTRODUCTION

Rocky Flats Plant (RFP)^a shipped solid, radioactive waste to Idaho National Laboratory (INL), located near Idaho Falls, Idaho. The waste was contaminated with minor amounts of weapons-grade plutonium, highly enriched uranium (HEU) (Oralloy), depleted uranium (DU), and toxic chemicals.

Shipping of waste began in April 1954 and continued into late 1989. Waste from RFP was deposited underground in a series of pits and trenches until 1970, when the U.S. Atomic Energy Commission (AEC) policy was implemented requiring segregation and retrievable storage of all solid transuranic (TRU) waste. After 1970, TRU waste received from RFP was placed in aboveground, earthen-covered retrievable storage. The aboveground stored waste was designated as TRU retrievable waste.

Because the definition of TRU waste changed in 1982, it is important to note that a large portion of the waste previously designated TRU is not TRU by today's definition. Originally, TRU waste was defined as all waste contaminated with TRU radionuclides in concentrations greater than 10 nCi/g (AEC 1973). However, in 1982, TRU waste was redefined based on a concentration of 100 nCi/g (DOE O 5820.1). Today, TRU waste is defined as waste material containing any alpha-emitting radionuclide with an atomic number greater than 92, a half-life longer than 20 years, and a concentration greater than 100 nCi/g at the end of the period of institutional control as defined in "Radioactive Waste Management Manual" (DOE M 435.1-1).

The waste from RFP was buried at the Subsurface Disposal Area, a radioactive waste landfill located in the Radioactive Waste Management Complex at INL. A baseline risk assessment and range of remedial alternatives are being developed under requirements of the Comprehensive Environmental Response, Compensation, and Liability Act (42 USC 9601 et seq., 1980) and associated *Guidance for Conducting Remedial Investigations and Feasibility Studies under CERCLA* (EPA 1988). In estimating risk and evaluating feasibility of various remedial alternatives, details of location and characteristics of radioactive waste in the landfill are essential.

The RFP shipping records, however, provided meager descriptions of waste shipped for burial. Likewise, INL receiving records for RFP buried waste were limited in characterization data. Consequently, INL personnel involved in developing the foundation for remedial decision-making initiated requests and inquiries to identify the types of RFP waste shipped and to document characterization data. These requests and inquiries were directed to Los Alamos Technical Associates, Inc., through a contractual arrangement. Los Alamos Technical Associates had several former RFP employees on their staff who were familiar with waste operations in the 1950s and 1960s and who had access to archival waste documentation that addressed shipping and characterization data. This report is a compilation of the data and information sent to various INL technical personnel. Consolidating these INL communications within this report provides a useful reference for present and future INL personnel.

a. Rocky Flats Plant is located 26 km (16 mi) northwest of Denver, Colorado. In the mid-1990s, it was renamed Rocky Flats Environmental Technology Site. In the late 1990s, it was again renamed to its present name, Rocky Flats Plant Closure Project. Most of the transuranic waste buried in the Subsurface Disposal Area originated at Rocky Flats Plant.

The report mainly concentrates on pre-1970 activities but also provides information related to post-1970 waste of interest. Appropriate background information is included to clarify the source and nature of the waste shipped. The following topics are addressed:

- Waste operations and facilities
- Waste characterization
- Waste containerization
- Waste assay
- Waste transportation and forms
- Special-order work
- Classified waste
- Special topics.

The majority of the report's text originated from documents, reports, memorandums, and other plant communications located in RFP archives. Supplemental information came from knowledgeable past RFP personnel.

Topics selected for this report were based on the author's judgment as to the data and information that INL personnel may find useful and on requests received from INL personnel. Many minor topics are not addressed that were judged of minimal importance and little consequence to remedial decision-making at INL.

Also included are a combined timeline of major occurrences at both RFP and INL, a list of correspondence from the author to Operable Unit 7-13/14 staff, a list of references cited throughout the report, and an extensive set of appendixes. The reference list contains sources that are readily available outside this report; the appendixes contain items such as reports, letters, and certificates that are not otherwise available. Each printed copy of this report will include a CD containing appendix content.

2. BACKGROUND

Construction of RFP started in 1951 and was completed by 1953. Plant operations began in 1953. Additional construction continued to meet the plant's changing War Reserve mission. The chief War Reserve mission was to fabricate components of nuclear weapons. The main products manufactured by RFP were the so-called triggers for thermonuclear weapons. At RFP, these plutonium triggers were referred to as pits for their resemblance to a fruit pit. A secondary mission was to fabricate and assemble special nuclear devices for testing at Nevada Test Site. Rocky Flats Plant also was assigned the task of disassembling obsolete returned pits to recover and recycle special nuclear material (SNM) components. The AEC selected Dow Chemical Company to operate RFP.

2.1 Rocky Flats Plant Facilities (1953–1969)

Rocky Flats Plant was originally organized and constructed based on radioactive materials processed and handled. Radioactive materials were weapons-grade plutonium, HEU, and DU.

Production facilities were the four plants listed in Table 1. Individual buildings within each plant, their operations, and materials processed also are included in Table 1.

During the 1950s, building numbers were composed of two digits such as Building 44, Building 71, and Building 81. Later, building numbers were changed to three digits. Consequently, the original two-digit building numbers became 444, 771, and 881 by placing the additional digit at the beginning of the building number. Waste shipping records for the 1950s used the two-digit building numbering system while later records employed the three-digit system. This report will use the three-digit building numbering system.

Initially (1953–1956), all plutonium operations were carried out in Building 771. In 1957, Buildings 776 and 777 were completed to accommodate plutonium technological changes and new pit designs.

Table 1. Rocky Flats Plant production plants 1953–1970.

Plant A – Manufacture nuclear weapon components of nonspecial nuclear material	
Materials –	Depleted uranium, depleted uranium alloys, aluminum, beryllium, stainless steel, copper, and other metals in minor amounts
Buildings –	444, 447, 883A, and 441
Operations –	Foundry, machining, heat treating, and inspection (444 and 447) Rolling and forming (883A) Analytical laboratory (441)
Plant B – Manufacture nuclear weapon components of highly enriched uranium (Oralloy)	
Materials –	Highly enriched uranium
Buildings –	881 and 883
Operations –	Foundry, machining, inspection, chemical recovery, and metal recycle (881) Rolling and forming (883B) Analytical laboratory (881)

Table 1. (continued).

Plant C – Manufacture nuclear weapon components of plutonium	
Materials –	Weapons-grade plutonium
Buildings –	771, 776, 777, 779, and 774
Operations –	Foundry (771 and 776) Machining (771 and 776) Inspection and assembly (777) Chemical recovery and metal recycle (771) Pyrochemistry (776) Liquid waste treatment (774) Research and development (779) Analytical laboratory (771 and 559)
Plant D – Pit assembly and certification	
Materials –	Plutonium, highly enriched uranium, depleted uranium, and other nonradioactive materials such as beryllium, stainless steel, and aluminum
Building –	991
Operations –	Assembly, inspection, certification, packaging, and shipping assemblies

New construction caused foundry and machining operations to be transferred to Building 776. Likewise, assembly and certification operations were transferred to Building 777. Vacated foundry and machining facilities in Building 771 were taken over by the Research and Development (R&D) Metallurgy Group. Vacated facilities in Building 991 were taken over by the Physics R&D Group.

Manufacturing of HEU components in Building 881 terminated in 1964; however, cleanout of HEU material continued for several more years. Rolling and forming operations in Building 883B were terminated also. The HEU area in Building 883B was converted to DU and beryllium operations. Vacated HEU areas in Building 881 were used for R&D projects and manufacturing of nonradioactive reservoir components.

Building 889 was built in the late 1960s to allow decontamination of process and machining equipment from Building 881 that had been contaminated with HEU. Most of the decontaminated equipment was reused by the plant maintenance group and the plutonium buildings.

With the addition of plutonium Buildings 776 and 777, the capability and capacity of the plutonium analytical laboratory located in Building 771 were inadequate for fast analyses of plutonium. Consequently, Building 559 was constructed in 1967 to expand plutonium analytical services to accommodate production demand. Building 559 became the primary laboratory for plutonium analyses.

In 1963-1964, expansion and upgrade of the plutonium chemical recovery systems in Building 771 were enabled by transferring support services—such as laundry, cafeteria, and offices—to new facilities next to the building. The laundry was moved to Building 778.

The plutonium upgrades and additions described above increased the quantity of plutonium-contaminated waste shipped to INL. The termination of HEU activities in the middle and late 1960s decreased the amount of HEU-contaminated waste shipped to INL.

Plant and production support groups contributed only a few drums per year to the waste sent to INL. The health physics laboratory in Building 123 generated mainly waste from bioassay and HEU.

The medical treatment facility in Building 122 handled cases of severe contamination by plutonium and HEU. Waste was usually transferred to Building 123 for disposal.

The general plant analytical laboratory was located in Building 441. The chief contaminant was uranium from DU and DU alloys. Natural thorium samples were analyzed occasionally for special projects. Building 441 generated a few drums of waste per year.

A special project, using natural thorium and DU, took place in Building 331 using temporary facilities. Several drums of thorium and DU waste were generated and shipped to INL.

Building 995 was the plant sanitation treatment facility. Occasionally, sanitation sludge would contain plutonium concentration levels requiring shipment to INL.

2.2 Additions to Rocky Flats Plant Facilities (1970–1989)

Nuclear weapon design changes and increased production demands from the Cold War caused expansion of production facilities at RFP. In addition, plutonium facilities constructed in the 1950s were almost 20 years old.

Building 707 was completed in 1970 to meet new pit designs that could not be manufactured in Buildings 776 and 777. In 1971, an annex was added to Building 707 to accommodate all of the operations in Buildings 776 and 777 as a result of the 1969 fire in the Building 776 foundry.

After cleaning up from the 1969 fire in Building 776, production operations were limited to special projects and disassembly of returned pits. The main focus of Building 776 became waste and residue treatment. A manual size-reduction facility was established in a previous plutonium storage vault as an outgrowth of the 1969 fire recovery operation. The size-reduction facility was a large generator of TRU waste sent to INL in the 1970s and 1980s for retrievable storage.

Building 444 remained a constant from 1970 until 1989 when DU and beryllium operations ceased; however, a production plating laboratory was built in 1981 on the second floor of Building 444. The electromachining and chemical milling processes generated a variety of liquid waste with high salt content.

With the advent of nondestructive assay (NDA) drum counters, a facility for drum counting and handling was built in 1971 between Buildings 771 and 774. This addition was designated as Building 771C and handled the majority of drum counting analyses for SNM.

Terminating HEU component manufacturing freed facilities in Building 881. Therefore, the analytical laboratory in Building 881 received the workload of the general plant analytical laboratory from Building 441. Building 441 was converted to offices for plant engineering, and DU waste was no longer generated in that building.

After 1970, pyrochemical procedures expanded. Building 776, housing pyrochemical production, was expanded to accommodate molten salt extraction (MSE) of Am-241 from returned pits. This facility generated spent salt, tantalum, and plutonium- and americium-contaminated waste from magnesium oxide crucibles.

To accommodate a real-time radiography unit, a second-generation NDA crate counter, and drum counters, Building 569 was built in 1987 and was ready for use in 1988. This facility generated very little TRU or other waste that was sent to INL.

New construction of Buildings 371 and 374—completed in 1980 and 1978, respectively—was justified by the age of Buildings 771 and 774 and expansion of pyrochemical processes in Building 776. Building 374 treated liquid waste from Building 371, effluent from the first precipitation stage from Building 774, and other plant-generated waste. Building 371 experienced technical difficulties in the chemical recovery of plutonium and was remodeled to accommodate the new accountability criteria for SNM. However, electrorefining of plutonium metal to a high purity level remained routine until 1989, and Building 374 successfully handled the majority of the plant liquid waste.

2.3 Beryllium Processing

Production of beryllium components began in 1957 and consisted of machining and inspection of beryllium forms supplied by offsite vendors. A wrought beryllium process was developed at RFP in the mid-1960s to recycle beryllium metal scrap into cast beryllium forms available for machining. While beryllium is not radioactive, it was often commingled with DU and other radioactive materials shipped to INL.

In 1975, offsite vendors began supplying beryllium blanks that required a minimal effort to machine into acceptable beryllium components. Consequently, the recycling and casting of beryllium at RFP ceased. The beryllium blanks provided by offsite vendors were composed of sintered beryllium, which contained 5–6% beryllium oxide. Eliminating the wrought process after 1975 significantly reduced the beryllium waste generated by Buildings 444, 447, and 883.

Beryllium components were handled and assembled into configurations in Buildings 707, 776, and 777. Returned pits were disassembled in Building 777. These activities generated trace amounts of beryllium in some of the waste shipped to INL.

Mixtures of beryllium and plutonium were processed in Building 771. The beryllium was dissolved in a mixture of nitric and sulfuric acid. The spent acid with the soluble beryllium was transferred to Building 774 for processing into a sludge for shipment to INL.

3. WASTE OPERATIONS AND ORGANIZATION

In January 1953, a waste disposal organization was established to supervise the ultimate disposal of processed liquid and solid waste and to gather and correlate disposal data. Initially called the Waste Disposal Unit, its title was changed to Waste Disposal Coordination Group to reflect its function more closely.

3.1 Waste Disposal Unit

The original staff was one full-time chemist with oversight by an analytical laboratory manager; however, by April 1954, the full-time staff increased to two with occasional support from a third employee from the general analytical laboratory. Waste was treated and packaged by operations personnel. The actual shipping was handled by the traffic group. The Waste Disposal Coordination Group did not have any facilities under its jurisdiction.

While staffing remained at two full-time employees, the workload increased significantly. By March 1954, 2,457 (30-gal) drums of solid waste had accumulated. An arrangement to ship solid radioactive waste to INL was authorized in April 1954 (see Appendix A). The first shipment to INL was made in April 1954 and was composed of 343 drums with a gross weight of 15,829 kg (34,896 lb).

The responsibilities of the Waste Disposal Coordination Group were the chemical, radiological, and physical states of the plant collection ponds and tanks; storage and disposal of contaminated waste; collection of waste data; and coordination of waste projects. This group also authorized the release of compliant wastewater from the plant site.

The Waste Disposal Coordination Group was attached to the analytical laboratory organization since its inception in 1953. In January 1965, the group was transferred to the Health Physics Group. In September 1970, the group's title was changed to Health Physics Waste Disposal. The staffing still remained at two full-time employees. In August 1971, the group title was changed to Waste Management Waste Disposal.

3.2 Waste Operations

In the early 1970s, safe disposal of radioactive waste became a national issue. In response, the AEC established new definitions of radioactive waste based on radiation levels. Two classes of radioactive waste pertinent to RFP were TRU waste and non-TRU waste. Because the definition of TRU waste changed in 1982, it is important to note that a large portion of the waste previously designated TRU is not TRU by today's definition (see Section 1). Upgrading the importance of radioactive waste to almost a product level and establishing rigid acceptance criteria for waste disposal placed a burden on plant operations.

In the mid-1980s, radioactive waste with a hazardous component was defined as mixed waste. The hazardous component was regulated by the Resource Conservation and Recovery Act (RCRA) (42 USC § 6901 et seq., 1976), while the radioactive component remained under jurisdiction of the U.S. Department of Energy (DOE). Consequently, part of the buried radioactive waste at INL could be designated as mixed waste.

To accommodate the increased emphasis on quality and packaging of radioactive waste, the Waste Operations Group was organized that included the original Waste Disposal Coordination Group. The Waste Operations Group still reported to the Health Physics Group.

The plant operating groups still packaged the solid radioactive waste but were required to comply with standard practices issued by the Waste Operations Group. Waste inspectors were provided by the Waste Operations Group to ensure compliance. Storing and loading of radioactive waste for offsite shipment were taken over by the Waste Operations Group. The liquid waste treatment facilities, Buildings 774 and 374, were transferred to the Waste Operations Group.

Operational experience, coupled with a significant enlargement of waste-associated activities and requirements, called for an independent waste operations group, which was formed in the late 1970s. The enlarged Waste Operations Organization consisted of a Solid Waste Operations Group, a Liquid Waste Operations Group, and a Waste Management Group. The Waste Management Group was responsible for the waste quality program, waste training, waste shipments, and recordkeeping.

4. WASTE CHARACTERIZATION

Rocky Flats Plant fabricated components of nuclear weapons from plutonium, HEU (Oralloy), and DU. These fabrication operations generated both liquid and solid contaminated waste. Liquid waste was either (1) aqueous-based solutions or (2) organic-based solutions. The solid waste consisted of the following five types:

- Type I—combustibles: paper, rags, wood, and plastics
- Type II—filter paper
- Type III—Chemical Warfare Service (CWS) filters
- Type IV—sludge
- Type V—noncombustibles: glass, brick, scrap metal, ceramics, and graphite.

These five types for solid waste were used from 1954 to 1970. (Section 4.2 describes an additional five categories [1 through 5], established mainly to describe generation of plutonium-contaminated waste, but also often employed to describe HEU-, DU-, and beryllium-contaminated solid waste. The five categories are not the same as shipping Types I through V above, although they are similar in some respects.)

Later, waste to be shipped was described and identified by item description codes. Machine coolants and other process liquids were filtered using filter paper that was classified as Type II waste. The use of filter paper declined in the 1960s, which significantly reduced this type of waste. Type III CWS filters refers to CWS filters that were used in building ventilation systems. The CWS filters were eventually replaced by high-efficiency particulate air (HEPA) filters. The Type IV sludge mainly refers to the series of sludge produced by the Liquid Waste Treatment Plant (Building 774). Other sludge in minor quantities came from process and cleanout building operations such as “still bottoms” (i.e., residue from distilling processes), degreasing bath residues, tank deposits, spent vacuum pump oil, and equipment maintenance.

4.1 Liquid Waste Treatment

The treatment of liquid waste accomplished four objectives:

- Removal of radioactive constituents from aqueous waste to allow solidification for disposal offsite
- Removal of chemical constituents from aqueous waste to satisfy drinking water standards and allow discharge offsite or reuse onsite
- Solidification of nonconforming aqueous waste for disposal offsite
- Solidification of organic liquid waste for disposal offsite.

Two liquid waste treatment plants—Buildings 774 and 374—were built at RFP. Building 774 was the initial plant and began operations in the 1952–1953 timeframe. Building 374 replaced the majority of Building 774 treatment operations, although organic liquid waste treatment remained in Building 774 along with support for Building 771. Building 374 came into use in the late 1970–1980 timeframe and did

not contribute to buried INL waste. Consequently, Building 374 treatment processes will not be addressed in this report.

The Liquid Waste Treatment Plant, Building 774, was built next to Building 771 to facilitate treating aqueous solutions generated by plutonium recovery operations in Building 771. Building 774 also treated radioactively and chemically contaminated aqueous waste generated by other plant activities. Consequently, Building 774 treated aqueous solutions contaminated with HEU, DU, and plutonium. The majority of aqueous waste solutions received for treatment were nitric-acid based. Plutonium aqueous waste solutions contained trace amounts of Am-241. Neptunium-237, U-233, and Cm-244 were received occasionally in trace-to-minor amounts from special-order projects. The major cations found in the waste solutions were aluminum, calcium, iron, potassium, magnesium, sodium, and silicon. The major anions were nitrate, sulfate, and chloride.

The contaminated solutions transferred to Building 774 were analyzed before being transferred either by pipeline or container for treatment. The SNM and DU content were the basis for normal operating loss estimated by the Nuclear Materials Management Group. Solutions received by pipeline were directed to designated receiving tanks based on their acidic, radiological, and chemical contents. The solutions received through drums and other containers were siphoned into receiving tanks or treated directly.

The treatment process in Building 774 used a two-stage, ferric hydroxide carrier precipitation process for radiolytic decontamination. To make the precipitating agent, ferric sulfate, calcium chloride, and a coagulating agent were added to a specific volume of water. The precipitating agent was then added to the acidic waste solution that was made basic (pH 11) with sodium hydroxide. Ferric ions combined with hydroxide ions to form a hydroxide floc that acted as a scavenger to remove radiolytical contaminants. Calcium ion overpowers any peptizing agents in waste solutions.

The neutralized solution with the precipitated slurry was pumped to a precoated rotary drum vacuum filter, which separated liquids from solids. The collected sludge was skimmed from the rotary drum filter through a knife-blade arrangement into a prepared drum for shipment offsite. This drummed sludge containing the bulk of the radioactive constituents was identified as first-stage sludge and designated as Series 741 sludge. The first-stage effluent was collected as feed for the second-stage precipitation. Plutonium waste solutions underwent two stages of precipitation while other plant waste solutions were treated through second-stage precipitation processes. Sludge collected from second precipitation was identified as second-stage sludge and designated as Series 742 sludge.

The filtrate effluent from the second-stage through the rotary vacuum drum filter was analyzed for its radiological and chemical content. If too high in radioactivity, the effluent was reintroduced to the second-stage precipitation process. If chemical content (mainly nitrate) was too high, the effluent was pumped to solar evaporation ponds, which were next to Building 774.

4.1.1 First- and Second-Stage Sludge

Sludge removed from the rotary vacuum drum filter was about 70 wt% water based on periodic analyses. Average concentration for first-stage sludge was 1.07×10^{-5} g/g or 3.48×10^{-5} Ci/g for americium and 4.71×10^{-5} g/g or 3.53×10^{-5} Ci/g for plutonium (see Appendix X).

First- and second-stage sludge was loaded into 17C or 17H steel drums of mostly 55-gal capacity, but at least 30-gal capacity. The maximum weights acceptable were 660 lb for 17H drums and 880 lb for 17C drums. Use of 17H drums was discontinued in favor of 17C drums for Building 774 sludge because of the higher maximum weight limit for 17C drums. The changeover started in the middle 1960s and was

completed by the late 1960s. A quantity of dry Portland cement was placed in the bottom of the drum. A polyethylene liner was positioned inside the drum. Additional dry Portland cement was interspersed with the filling sludge. After sealing the liner, additional dry Portland cement was placed on top of the liner. The filled drum was sealed, weighed, labeled, logged, and surveyed for surface contamination and external radiation levels.

Occasionally, first- and second-stage sludge was mixed to meet certain shipping requirements. This type of sludge was designated as Series 7412 sludge. Building 774 was expanded with an addition that was designated as 74A. Starting up the organic sludge processing unit located in the 74A addition produced experimental sludge drums that were identified as 74A sludge drums. Later, this sludge product was designated Series 743 sludge.

4.1.2 Off-Specification Waste Solutions

Aqueous waste solutions that did not meet feed specifications for first- and second-stage treatment were processed directly. These waste solutions contained objectionable constituents such as complexing agents, hazardous chemicals, and certain radioactive isotopes not normal to the plant. High chloride solutions, such as hydrochloric acid solutions, were also candidates for this type of treatment. These solutions were solidified directly with Portland cement. Acidic solutions were made basic before adding the cement.

Special solidification drums were prepared using a mixture of Portland cement and an absorbent material. The solidification drum was connected to the solidification glove box through an O-ring drum liner arrangement. The basic waste solution was added to the prepared solidification drum; the Portland cement then reacted with the added solution to form a solid. The added absorbent material aided distribution of the waste solution within the drum. A maximum of 94.6 L (25 gal) of waste solution could be solidified per drum. Waste solution received in small volumes (bottle containers) was often treated directly and placed in a prepared drum. The disposition of off-specification waste was based on quantities received and operating experience. Off-specification waste was designated as Series 744 sludge, sometimes referred to as special setups.

4.1.3 Evaporator Salts

Treated solutions high in chemical salts but meeting radioactive levels were stored in solar evaporation ponds next to Building 774. Consequently, an evaporator system was added to Building 774 composed of an evaporator, a double drum dryer, a dust scrubber system, and a steam condensate collector. A steam-heated heat exchanger was employed as the heat source for the evaporator. Water vapor generated by the evaporator was exhausted to the atmosphere through baffles and entrainment separation pads. Concentrated salt liquid from the evaporator was transferred to the steam-heated double-drum dryer. Remaining water was removed, leaving a film of dry salts baked on the rotating drum surfaces, which were then scraped using a knife-blade arrangement. The salts were collected in a catch container that was weighed and emptied into a wooden crate for shipment offsite. Evaporator salts, also called nitrate salts, were designated as Series 745 sludge.

4.1.4 Contaminated Drums

Contaminated empty drums were shipped in crates and designated as Series 746 sludge. These drums were rinsed with an appropriate solvent to reach a contamination level of <3 g of plutonium.

4.1.5 Treatment of Organic Liquid Waste

Organic liquid waste was composed mainly of a variety of oils and solvents. The types of oils received for treatment were basically cutting, lubricating, hydraulic, and vacuum pump oils. Solvents were used as degreasing and cleaning agents. Several organic liquids were employed for density measurements on machined parts. The analytical and R&D laboratories generated small volumes of contaminated organic liquids with a variety of extraction agents. The largest contributor to organic liquid waste was spent lathe coolant generated by plutonium machining operations. The majority of waste solvents were basically chlorinated and fluorinated hydrocarbons. Plutonium lathe coolant was diluted with carbon tetrachloride (CCl_4) to 30% oil and 70% CCl_4 . However, the percentage of CCl_4 remaining in spent lathe coolant received in Building 774 was reduced by evaporation and probably ranged between 25 and 60%.

Organic liquid waste was treated by mixing the organic liquid with an absorbent powder to form a greaselike substance. Consequently, this operation was referred to as the “Grease Plant” or “Jelly Factory.” Microcel, manufactured by Johns-Mansville, was the absorbent material used and was mainly calcium silicate. The mixing ratio was 45 kg (99 lb) of Microcel (three bags) to 190 L (50 gal) of organic liquid. Microcel obtained from other manufacturers did not mix very well. Therefore only Microcel from Johns-Mansville was used.

Mixing was accomplished by a blender (Readco Processor) that was enclosed within a glove box. Organic liquid and absorbent powder were piped into the mixer at controlled rates. On completing the blending process, the resulting greaselike mixture was discharged into a shipping drum attached to the glove box through an O-ring attachment. Loaded drums were sealed, weighed, labeled, logged, and surveyed for surface contamination and external radiation.

In the middle 1980s, the Grease Plant treatment of contaminated organic liquid waste was replaced with an improved solidification process identified as Organic Accelerated Solidification and Immobilization System (OASIS). This process is relevant to retrievably stored TRU waste, but not to buried waste. The constituents and their respective weights required to prepare the solidification medium for a given 55-gal drum are listed in Table 2.

Table 2. Constituents and weights for a 55-gallon drum.

Constituent	Weight (lb)
Contaminated oil	170
Emulsifier	25
Envirostone	250
Water	42
Total	487

Anderson et al. (1985) provide a more thorough description of the liquid waste treatment operations in Buildings 774 and 374.

4.2 Solid Waste Treatment

The five categories listed below were established mainly to describe generation of plutonium-contaminated waste, but also were often employed to describe HEU-, DU-, and beryllium-

contaminated solid waste. (Previously, the five Types I through V of waste shipped to INL were described in Section 4, “Waste Characterization.” These descriptions were used by RFP to generally describe the types of waste shipped per container. However, RFP also identified waste by generation categories for operational purposes, waste generation statistics, and cost distribution. The five categories below are not to be confused with the shipping Types I through V although they are similar in some respects.) The five categories include:

1. Line-generated
2. Sludge
3. Filters
4. Maintenance operations
5. Non-line-generated.

4.2.1 Line-Generated Waste

Line-generated waste was produced by glove-box operations. This waste usually was highly contaminated with plutonium and required a plutonium assay to determine disposition status. If above an established economic discard limit, the waste was designated as recoverable residues; however, the majority of line-generated waste was composed of items used in operating and maintaining the line.

Contaminated items were placed in a drum that was attached to the glove-box line. The drum was equipped with a drum liner. When full, the drum was disconnected, sealed, labeled, weighed, surveyed for contamination, and transferred to storage to await assay. All line-generated waste was segregated according to waste Types I through V (see Section 4).

4.2.2 Sludge Waste

Liquid waste treatment processes carried out in Building 774 produced the majority of sludge waste. However, occasionally, contaminated sludge accumulated within a piece of processing equipment and was designated as a sludge waste according to the building where it was generated, such as Buildings 771, 776 or 777, 881, and 444 or 447.

4.2.3 Filter Waste

Filter waste refers mainly to ventilation filters used to remove airborne contamination. Large filters ($2 \times 2 \times 1$ ft) were used in the exhaust plenum systems, and small filters ($12 \times 12 \times 8$ in.) were used in intake and exhaust systems on glove boxes. These filters were assayed to determine whether they were above or below economic discard limits. If above discard limits, the filter medium was removed and processed to recover SNM; however, filters were not assayed and processed until the 1960s. Therefore, filters disposed of earlier may have contained concentrations of SNM higher than the economic discard limits.

4.2.4 Maintenance Operational Waste

Most contaminated waste generated by maintenance operations consisted of contaminated equipment and ancillary electrical and piping apparatus. A significant amount of this waste was packaged in wooden crates for shipment to INL.

Glove-box maintenance required enclosure by a plastic tent to control contamination. On completion of maintenance, these plastic tents were packaged as Type I waste.

4.2.5 Non-Line-Generated Waste

Non-line-generated waste was produced in process areas outside of the glove-box lines. This waste consisted of rags, absorbent wipes, surgical gloves, and other small, routinely used items. A significant amount of this waste was generated by housekeeping activities and had trace amounts of contamination.

4.3 Isotopic Levels in Waste

The four most prevalent types of radiological elements shipped to INL were: (1) weapons-grade plutonium, (2) HEU, (3) DU, and (4) Am-241. Americium-241 is the daughter product of the beta decay of Pu-241. The isotopic content of weapons-grade plutonium varied slightly from year to year as the mixture of returned plutonium and new plutonium from the Hanford and Savannah River Site reactors was not constant (see Table 3 for variations in the plutonium isotopic concentrations from 1959 to 1976). Improved mass spectrometry instrumentation provided lower detection limits for Pu-238 and Pu-242, providing values below 0.05 wt% rather than just a minimum of 0.05 wt%. This gave a more definitive evaluation of the isotopic content in the waste. See Table 4 for typical isotopic profiles for DU, HEU, and weapons-grade plutonium.

Table 3. Rocky Flats Plant plutonium isotopic levels in waste (stream averages—plutonium wt%).

Calendar Year	Pu-238	Pu-239	Pu-240	Pu-241	Pu-242
1959–1960	<0.0500	93.714	5.593	0.5932	<0.0500
1961–1962	<0.0500	93.817	5.486	0.5979	<0.0500
1963–1964	<0.0500	94.398	4.854	0.6482	<0.0500
1965–1966	<0.0500	93.586	5.823	0.5610	<0.0500
1967–1968	<0.0500	93.451	5.953	0.5670	<0.0500
1969	<0.0500	93.538	5.953	0.4790	<0.0500
1970	<0.0500	93.450	5.965	0.4850	<0.0500
1971	<0.0500	93.533	5.929	0.4380	<0.0500
1972	<0.0500	93.513	5.939	0.4480	<0.0500
1973	<0.0500	93.596	5.918	0.4300	<0.0500
1974 (first half)	<0.0500	93.571	59.000	0.4855	<0.0500
1974 (second half)	0.0104	93.656	5.893	0.4620	0.0317
1975	0.0102	93.707	5.861	0.3940	0.0266
1976	0.0102	93.827	5.814	0.3510	0.0219

Table 4. Typical isotopic concentrations in waste.

Material	Isotope	Weight Percent
Depleted uranium	U-235	0.30
	U-238	99.70
Highly enriched uranium (Oralloy)	U-234	1.02
	U-235	93.17
	U-236	0.44
	U-238	5.37
Weapons-grade plutonium	Pu-238	0.01
	Pu-239	93.63
	Pu-240	5.97
	Pu-241	0.37
	Pu-242	0.02

4.4 Waste Constituents

Previous discussion indicated that waste shipped to INL before 1970 was characterized by five types of materials. This same characterization will be followed in discussing waste constituents.

4.4.1 Combustible Waste—Type I

Contaminated combustible waste consisted mainly of rags, paper, plastics, wood, tape, rubber, and contaminated clothing, as described below.

Rags—Cotton-based rags were used mainly to wipe up spills and for cleaning purposes.

Wood—The major wood item was contaminated forklift pallets. Wood planking used in maintenance operations also contributed to the wood category.

Tape—Yellow vinyl tape and white masking tape were used extensively for closing plastic bags, for erecting plastic working tents (houses) for maintenance, and for many other applications.

Plastics—Polyvinyl chloride (PVC) and polyplastics were the chief plastic materials. Major plastic items were bags, tubing, and sheet forms. Tygon tubing was another form of plastic discard.

Paper—Paper items included absorbent wipes, laboratory filter paper, contaminated forms, packing paper, paper cartons, and miscellaneous paper articles.

Rubber—Major rubber items were surgical gloves and glove-box gloves. Minor rubber items were rubber stoppers, gaskets, and tubing.

Contaminated clothing—The majority of protective clothing was manufactured from cotton. Routine protective clothing consisted of coveralls, t-shirts, shorts, socks, surgical caps, and an occasional outside jacket. Cotton gloves were used extensively. Discarded booties (cotton canvas tops and rubber bottoms) were included in the discarded clothing.

Radioactive and hazardous material garments—Maintenance operations and decontamination efforts often required special protective garments and supplied breathing air for contact personnel. For contamination control, plastic tents (houses) were constructed to enclose the operational area. See Table 5 for a listing of these items.

Table 5. Personal protection garments used in working with radioactive and hazardous materials.

Date	Article	Material (description)	Use
1961 to early 1980s	Body suit	Tyvek (white)	Radiation worker
1961 to early 1980s	Hood	PVC (transparent)	Radiation worker
1961 to present	Gloves	Rubber (surgeon gloves)	Radiation worker
1961 to present	Gloves	Butyl (arm length)	Radiation/hazardous environments
1961 to early 1980s	Body suit	PVC (yellow rain suit)	Radiation/hazardous environments
1961 to present	Tape	Vinyl (yellow), masking (white)	Radiation/hazardous environments
Early 1980s to 1992	Encapsulated body suit with removable hood (Level A suit, SBAG)	Vinyl Technologies Corp., vinyl suit (orange), ABS valves (black), press-polish transparent vinyl hood	Radiation/hazardous environments
Early 1980s to present	Gloves	Rubber (orange, anticontamination clothing)	Radiation/hazardous environments
1992 to mid 1990s, alternating with Rich Industries Level B garment	Encapsulated body suit and hood (Level B garment)	Vinyl Technologies Corp., two films PVC with polyvinylidene fluoride (white), ABS valves (black), transparent vinyl hood	Radiation/hazardous environments
1992 to mid-1990s, alternating with Vinyl Technologies Corp. suit currently used at Rocky Flats Environmental Technology Site since 1995	Encapsulated body suit with hood (Level B garment)	Rich Industries, Sarenex/Tyvek body (white), vinyl valves (black), transparent vinyl hood	Radiation/hazardous environments
1961 to 1997	Tent (contamination control barrier)	Polyethylene (semitransparent) 6-8-ft widths	Radiation/hazardous environments

PVC = polyvinyl chloride

4.4.2 Filter Paper—Type II

In the 1950s, filter paper was used to remove particulates from lathe coolant and cutting oils. No significant information was located addressing this type of waste; however, the amount of Type II waste was minimal compared to the other four types. Filter paper was discontinued eventually in favor of other forms of machining filters, although Type II was used occasionally when warranted.

4.4.3 Chemical Warfare Service Filters—Type III

The original ventilation and glove-box filters used in the 1950s and 1960s were basically CWS filter designs. Waste designated as Type III indicated filters employed in ventilation systems and other air-filtered streams.

4.4.4 Sludge—Type IV

The bulk of sludge shipped to INL was generated by Building 774, the Liquid Waste Treatment Facility. However, equipment cleanouts in other buildings occasionally produced sludge-based material, which was designated as Type IV waste (see Section 12.19 for a description of sludge).

4.4.5 Noncombustible Waste—Type V

Noncombustible waste consisted of scrap metal, obsolete processing equipment, broken tools, glove boxes, ventilation ducts, piping, electrical wiring, lighting fixtures, and other metallic objects. Maintenance operations generated the majority of Type V waste. Other Type V waste forms were contaminated soil, concrete, macadam pavement, ceramics, glass, and graphite molds.

4.4.6 Waste from Buildings 444, 447, and 883

Waste generated by Buildings 444 and 447 was contaminated with DU and beryllium. Typical beryllium-contaminated waste included broken obsolete graphite molds and crucibles, tool bits, chucks, coolant, filters, sweepings, absorbent wipes, and other miscellaneous items. Large amounts of sandpaper and emery cloth were discarded as combustible waste (Type I). Work tables for coating molds were covered with butcher paper, which was discarded as combustible waste.

In addition to straight DU fabrication, several DU alloys were used in fabrication operations. The two main alloys were DU-niobium and DU-titanium. Niobium and titanium concentrations ranged from 1 to 6 wt% in the alloys. Consequently, DU-contaminated waste contained a combination of DU and DU alloys.

Depleted uranium alloys employed during the 1953–1979 and 1980–1989 timeframes are arranged in descending order of use with number 1 being the largest amount of DU material used.

Depleted uranium alloys employed during 1953–1979 include:

1. Unalloyed DU
2. Uranium-titanium alloys (U-0.75 wt% titanium)
3. Uranium-niobium alloys (U-2 wt% niobium) (U-6 wt% niobium)
4. Uranium-molybdenum alloys (U-2 wt% molybdenum) (U-4 wt% molybdenum)
5. Uranium-miscellaneous; R&D efforts and special-order work, no large quantities involved (U-1 wt% molybdenum—0.75 wt% titanium—1 wt% niobium and U-1 wt% niobium—0.5 wt% titanium—1 wt% zirconium).

Depleted uranium alloys employed during 1980-1989 include:

1. Unalloyed DU
2. Uranium-niobium alloy (U-6 wt% niobium).

Aluminum, copper, and lead were used as substitutes for DU in casting and back-machining operations. Roaster oxide (i.e., uranium chips, fines, and chunks oxidized in a furnace to uranium oxide) was a large contributor to the waste stream. Graphite molds and crucibles too large to fit into a 55-gal drum were placed in 4 × 4 × 7-ft crates. Mold coatings employed were yttrium oxide, niobium oxide, and aluminum oxide suspended in water, glass, and calcium fluoride.

Buildings 444 and 447 developed their own waste identification codes to segregate the waste. These letter codes were included sometimes on shipping load lists. See Table 6 for a list of these letter codes.

Table 6. Waste codes for Buildings 444 and 447.

Code	Description	Shipping Type
A	Filter paper	II
B	Coolant still bottoms	IV
C	Metal fire brick	V
D	Paper, rags, wood	I
E	Waste oil	N/A ^a
F	Graphite	V
G	Perclene still bottoms	N/A ^a
K	Process waste filter	IV
M	Cyanide cement	V
N	Miscellaneous solids	V

a. Not applicable. Waste was processed onsite and not shipped.

Any equipment or apparatus that moved or displaced air had a CWS or a HEPA filtering system. The exhaust from furnace vacuum pumps was filtered also. These spent filters were disposed of as Type III waste.

The vacuum furnaces were lined with metal bricks. These bricks were contaminated with DU and were discarded as Type V waste.

Protective clothing was provided to operating personnel. Discarded contaminated clothing included aprons, cotton gloves, asbestos blankets and gloves, hoods, jackets, coveralls, shin guards, and underclothing. These items were discarded as Type I combustible waste.

Fabrication processes generated a variety of oil-based and organic solvent waste. The majority of the oil waste was burned on plant site. The solvent waste was distilled for reuse when applicable. Still bottoms were sent to Building 774 for disposal through the Grease Plant, resulting in Series 743 sludge.

Aqueous-based lathe coolants were used with DU machining operations for fire prevention. These spent coolants were transferred to Building 774 for disposal by the second-stage precipitation process, resulting in Series 742 sludge.

Building 883 augmented the fabrication operations in Buildings 444 and 447. The DU ingots were received from Building 444 for rolling and forming in Building 883 Section A. Ingots were placed in a furnace or in a heated eutectic salt bath to prepare them for rolling. After rolling into sheet form, the sheets were annealed in a second salt bath. Appropriate shapes were cut from the annealed sheet for forming contoured parts that were returned to Building 444 for final machining operations.

Sheet trimmings and other DU residues were returned to Buildings 444 or 447 for recasting or for conversion to roaster oxide for shipment to INL. Cleaning solvents such as trichloroethylene (TCE) and tetrachloroethylene (PCE) were used in processing operations.

Spent TCE, PCE, and chlorofluorocarbons were sent to Building 774 for disposal through the Grease Plant. Spent salt baths with uranium oxide were packaged into drums and shipped to INL as Type IV sludge. Although Dow Corning 550 fluid was occasionally used as a cooling bath after being heat treated and sent to the Grease Plant in Building 774 for disposal, such disposal was infrequent.

Spent salt baths consisted of a mixture of sodium, potassium, and lithium carbonate contaminated with uranium oxide. The original salts were a white crystalline powder, but spent salts were colored by the spent oxides and turned grayish or blackish. The spent salt baths were packaged into drums and shipped to INL.

Building 883 Section B was dedicated to rolling and forming HEU items. The process employed was the same as described for DU, except that the original ingot came from Building 881. Spent salt baths were sent to Building 881 for recovery of HEU metal fines and oxide. Resulting combustible waste also was sent to Building 881 for incineration and subsequent recovery of HEU from the ash.

In 1965, HEU operations were terminated in Building 881 and 883, except for cleanup activities. Building 883 Section B remained idle until 1983, when fabrication of DU armor plate began for the U.S. Army. This operation continued into the 1990s.

In 1962, beryllium-forming processes were established in Building 883 Section A and continued until the mid 1980s. Beryllium ingots were cast in Building 444 and encapsulated in steel cans, which were heated and rolled into a sheet form. The steel can container was then cut away to remove the rolled beryllium sheet. The sheets were etched with acid—to remove microcracks and for thinning—in a bath of combined nitric and hydrofluoric acids. After etching, the sheets were heat treated in either an acid bath or a eutectic salt bath or both. Desired shapes were then cut and formed from the sheet. The formed beryllium part was sent to Building 444 for final machining.

The steel can cuttings were disposed of as noncombustible waste (Type V) that was shipped to INL. The spent acid baths were transferred to Building 774. In the late 1970s and 1980s, the spent acid baths were transferred to Building 374, the new Liquid Waste Treatment Plant. The spent eutectic salt baths were packaged into drums and sent to INL for retrievable storage.

The use of chlorofluorocarbons was discontinued in 1988. The PCE and TCE usage also was discontinued in favor of water and Oakite in the 1985–1986 timeframe. Initially, one of the large metal presses employed an oil that contained polychlorinated biphenyls. The oil was removed from the press and deposited in the polychlorinated biphenyl storage areas; however, any oil leaks from the press were probably absorbed onto rags or absorbent wipes, which were part of the combustible waste. The amount of this type of combustible waste was minimal.

Building 883 occasionally fabricated items from tantalum, titanium, stainless steel, cadmium, and aluminum. The majority of the generated scrap was collected and sold to offsite vendors or reused on

plant site. Any combustible waste with trace amounts of the above metals was mixed with routine-generated combustible waste.

4.4.6.1 Graphite Mold Failures. Occasionally, a mold coating would fail, and the molten beryllium or DU would react with the graphite and produce a hole. The molten metal would puddle in the bottom of the furnace through the hole in the graphite mold. Cleanup would include the melt, fire brick, graphite crucible, and all of the cleanup materials. These materials were consolidated into their own drums or boxes with no dilution from other materials. The DU was written off as a normal operational loss because it was an accountable material.

4.4.6.2 Rejected Depleted Uranium Slabs. Recast DU scrap produced an ingot $20 \times 24 \times 2$ in. that was called a slab. If the slab failed to meet the impurity specifications, it was discarded as waste. The slab was placed in a drum and surrounded by soft waste to secure the slab within the drum. Although the slab was listed as Type V, substantial quantities of Type I waste were also in the drum.

4.4.6.3 Asbestos Items. Asbestos items were very common in the Building 444 foundry. The asbestos items in the following list were used in foundry operations and discarded as waste—either Type I or Type V depending on the operator's discretion—when contaminated:

- Aprons
- 4×8 -ft fire blankets
- Gloves
- Jackets
- Hoods
- Shin guards
- Tape.

4.4.6.4 Spill Cleanup. The foundry used many large cooling water systems whose cooling water contained a chromate inhibitor. Cooling water that occasionally leaked was cleaned using rags and absorbent wipes and discarded as Type I waste.

The foundry also used large vacuum systems that required significant amounts of vacuum oils. Oil changes and leaks often generated Type I waste.

4.4.6.5 Miscellaneous Waste Materials. The following is a list of materials that were discarded as waste in trace and minor quantities:

- Grinding wheels and motors
- Unclassified tooling
- Cadmium plating turnings—back machining
- Chromium plating turnings—back machining
- Lead casting residues—skull and turnings
- Aluminum chips, turnings, and casting skull
- Copper turnings and casting skull

- Spent furnace fire brick
- Contaminated furniture.

4.4.6.6 Waste Segregation. Building 444 had a beryllium machine shop, a DU machine shop, and a foundry that accommodated both beryllium and depleted uranium. The waste generated by the machine shops was segregated, but foundry waste could be commingled. Although drums and boxes generated were not marked always as to their origin within Building 444, a commingled drum was always identified as a beryllium drum.

4.4.7 Building 881 Waste

The mission of Building 881 was to fabricate weapon components of HEU (Oralloy). Building 881 had the capabilities of a foundry, of machining and inspection, and of chemical recycling. The HEU residues and metal scrap were processed to recover uranium and produce pure uranium metal. The majority of combustible waste was incinerated, and HEU was recovered from the ash. Fabricating components of HEU began in 1953 and ended in 1965. Cleaning and removal of equipment continued into the late 1960s.

The HEU waste generated from operations was mainly graphite molds and crucibles. Magnesium oxide molds and crucibles were used initially (1953–1955) but were replaced by graphite molds and crucibles. Consequently, the noncombustible Type V waste shipped in 1954–1955 to INL could contain magnesium oxide crucibles.

A secondary waste item was formed when impure materials contaminated with HEU were ground to a fine powder, leached in nitric acid, filtered, and collected as solids. These solids were called mud, which was then dried, assayed, packaged into drums, and shipped to INL as Type IV sludge. Discarded nitric acid solution with trace amounts of HEU was transferred to Building 774 for second-stage processing or to the solar evaporation ponds. Initially, these waste solutions were set in concrete that was sent to INL. Removing HEU equipment in the late 1960s and early 1970s contributed to Type V noncombustible waste.

Beginning in the early 1960s, plutonium surface contamination on HEU components was removed in Building 881. The HEU units were sprayed with nitric acid to remove the plutonium and then washed with water to remove residual acid. Plutonium-free units were cut up for introduction to the casting process. After 1965, the HEU components were sent to the Oak Ridge National Laboratory (ORNL) Y-12 plant.

The leach solution collected from sprayed nitric acid was concentrated by evaporation and uranium and plutonium precipitated by adding ammonia. The precipitate was dried and calcined to an oxide. If the oxide was very low in plutonium (<1 ppm), the oxide was shipped to the ORNL Y-12 plant. Oxides above the plutonium limit were shipped to Savannah River Site. This process continued until 1974, when it was transferred to Building 771. The oxide generated from the Building 771 process was sent to the Idaho Chemical Processing Plant (now Idaho Nuclear Technology and Engineering Center) at INL in the 1980s, provided the plutonium content was <500 ppm.

Shell Vitara oil was the machining coolant employed in Building 881 and was circulated through a centralized system. Spent oil was filtered to remove any uranium fines and then packaged in drums for disposal. A limited quantity was burned, but the majority was processed through the Grease Plant in Building 774. Other degreasing solvents such as TCE and PCE were transferred also to the Grease Plant for offsite disposal.

Several special projects were carried out in Building 881. Neptunium-237 was introduced into HEU and DU components as a bomb fraction tracer for underground testing at the Nuclear Test Site. The U-233 components were fabricated for tracing purposes and other nuclear experiments. Both Np-237 and U-233 were received as oxides and converted to metal for casting. Waste generated was mainly graphite molds and crucibles and combustible waste. Combustible waste was not incinerated but shipped to INL as Type I waste.

Thorium-containing components were fabricated for a short time in the late 1950s to early 1960s. Scrap and residues were shipped to either Savannah River Site or ORNL. The only waste sent directly to INL was commingled combustible waste.

Building 881 had an analytical laboratory for control purposes. Wet chemical analyses and emission spectrographic analyses were performed on HEU products and residues. Waste generated was mainly combustibles and a few drums per year of noncombustibles. Chief noncombustible items were emission spectrographic graphite electrodes and spent laboratory equipment.

In the late 1960s, Building 881 was converted to fabricating stainless steel reservoirs. Waste generated by this operation was not sent to INL.

4.4.8 Buildings 122 and 123 Waste

Building 122 was a medical facility, which treated industrial injuries, decontaminated personnel, and carried out routine physicals. Decontamination activities generated discarded combustibles that were shipped to INL at a level of a few drums per year.

Building 123 was a health physics laboratory, which carried out bioassays and engaged in low-level radiological studies. These efforts generated a few drums per year of combustible waste that was shipped to INL.

4.4.9 Building 991 Waste

Building 991 was originally constructed to carry out three functions: (1) shipping and receiving SNM, (2) storing SNM, and (3) assembling nuclear weapon components. Assembly operations were discontinued in 1957 and transferred to Building 777; however, a few components were assembled in the 1960s on a special basis. Building 991 continued to serve as a shipping, receiving, and storing facility for SNM into the 1990s. In addition, the building housed a metallurgical laboratory and other R&D laboratory facilities.

Functions carried out in Building 991 generated mainly combustible waste contaminated with trace amounts of HEU, DU, beryllium, and occasionally plutonium. Limited quantities of degreasing and cleaning solvents also were used. The metallurgical laboratory employed small amounts of isopropyl alcohol, carbide grinding paper, metal etching solutions, and nonhazardous polish solutions. Waste generated included cutting fines from sample preparation that were processed on plant site if the fines were HEU, DU, or plutonium.

The quantity of waste generated by Building 991 and shipped to INL was limited to less than 100 drums per year during the 1950s and less than 50 drums per year from 1960 to the end of production.

4.4.10 Building 886 Waste

Building 886 was essentially a laboratory facility constructed for nuclear criticality experiments pertinent to RFP HEU and plutonium operations. The chief fissile material was 93 wt% HEU in various forms; plutonium oxide in limited amounts was available but not used. Building 886 contributed to waste shipped to INL during the 1960s, 1970s, and 1980s. The main contributor was combustible waste contaminated with HEU. The facility generated less than 50 drums per year.

4.4.11 Sewage Treatment Plant—Building 995

Occasionally, sludge from the sewage treatment plant (Building 995) became contaminated with trace amounts of plutonium. When this occurred, the sludge was packed into crates or drums and shipped to INL in the 1950s and 1960s. The amount shipped was small in comparison to sludge shipments from Building 774.

4.4.12 Building 331 Waste

Building 331 was a temporary development facility engaged in evaluating equipment and methods applicable to casting and fabricating DU items. The facility also carried out a very limited thorium project. Waste generated was mostly DU contaminated and was limited to less than 100 drums shipped to INL.

4.4.13 Building 865 Waste

Building 865 was an R&D metallurgical facility constructed to develop and evaluate equipment and procedures associated with foundry, forming, swaging, and machining of DU, DU alloys, beryllium, and other metals of interest to the War Reserve. The facility came into use in 1970 and did not contribute to TRU waste sent to INL; however, Building 865 may have contributed a small amount to 1970 waste sent to INL that was contaminated with DU and beryllium.

4.4.14 Plutonium Waste-Generating Facilities

The facilities that generated most of the plutonium waste shipped to INL in the 1950s were Buildings 771, 774, 776, and 777. Waste from these buildings continued to be shipped until INL terminated shipping from RFP in 1989. Building 779 (R&D facility) and Building 559 (analytical laboratory) began shipping plutonium waste in the latter part of 1960 and continued until termination of shipping from RFP in 1989. These buildings in which plutonium was worked were the main contributors to buried pre-1970 waste sent to INL. In 1958, Building 778 was constructed next to Building 776 and provided laundry services to all the buildings in which plutonium was worked.

Additional contributors from 1970 until INL shipping termination in 1989 were Building 707 (fabrication and pit assembly), Building 371 (chemical recovery and pyrochemistry), and Building 374 (liquid waste treatment). Waste generated by these facilities included all waste Types I through V and, later, all kinds of waste labeled with item description codes.

Boron glass raschig rings, which resemble napkin rings, were used for nuclear criticality safety in these plutonium facilities in tanks and other containers holding liquid. The rings were replaced because of sludge buildup or failed dimensional testing. The failed rings were leached with nitric acid, rinsed in water, and packaged for shipment to INL. Leached raschig rings made up a significant amount of glass sent to INL.

Raschig rings were used also in Building 881, which processed HEU. Cleaning out Building 881 contributed to the raschig ring waste in the Subsurface Disposal Area.

4.4.15 Offsite Waste Sources

Offsite educational institutions, private companies, and other federal agencies called on RFP to assist with disposal of their radioactive waste. Offsite sources usually were under contract to the AEC for some type of work. In June 1957, the RFP AEC office granted permission for the Dow Chemical Company to accept radioactive waste at RFP that was generated by local offsite companies, institutions, and government agencies. The driving reasons for granting this permission were the capability and capacity of RFP to accept waste and ship it to federally approved facilities. Consequently, RFP accepted and shipped radioactive waste received from offsite sources from 1957 to 1971. Solid waste received was not treated or repacked at RFP, but only shipped through to INL.

The Coors Porcelain Company had a contract to produce reactor components for the TORY II-C reactor through a contract with the Lawrence Radiation Laboratory at Livermore, California, in the early 1960s. Beryllium- and uranium-contaminated liquid waste was put in solar evaporation ponds. Solids recovered from the solar evaporation ponds were eventually shipped to INL.

A summary of waste received and its corresponding shipper is shown in Table 7. Information provided in Table 7 was derived from monthly history reports issued by the Waste Disposal Coordination Group, RFP, and *Rocky Flats Toxicologic Review and Dose Reconstruction Task 3&4 Report* (ChemRisk 1992).

Descriptions of waste received from offsite sources could not be located; however, two letters were located indicating that two drums received from the Colorado School of Mines contained soil samples contaminated with plutonium (see Appendix B).

Table 7. Offsite shipments received by Rocky Flats Plant and shipped to Idaho National Laboratory.

Calendar Year	Shipper		Description
1957	Martin Aircraft	5	55-gal drums
	Lowry Air Force Base	51	cartons
	U.S. Bureau of Reclamation	2	55-gal drums
1958	Lowry Air Force Base	64	cartons
	Sunstrand	29	55-gal drums
1959	Sunstrand	10	55-gal drums
	Lowry Air Force Base	2	55-gal drums
1960	Denver Research Institute	3	Chemical Warfare Service filters
1961	G. E. Sandia	28	20-gal drums
		13	15-gal drums
	Denver Research Institution	4	55-gal drums
		2	30-gal drums
		1	carton
	Lowry Air Force Base	4	55-gal drums
	Sunstrand	2	30-gal drums

Table 7. (continued).

Calendar Year	Shipper	Description	
1962	Coors Porcelain Company	99,700 gal of beryllium-contaminated liquid waste to solar ponds ^a	
	G. E. Sandia	9	20-gal drums
	Denver Research Institute	7	cartons
	Coors Porcelain Company	137,000 gal of beryllium-contaminated liquid waste to solar ponds—first uranium-contaminated waste received ^a	
	Colorado University Medical School	First contaminated waste received	
1963	Lawrence Radiation Laboratory	42	55-gal drums
	Coors Porcelain Company	22,000 gal of beryllium-contaminated liquid waste to solar ponds ^a	
1964	Colorado University Medical School	3	55-gal drums
	Colorado School of Mines	2	55-gal drums
	Coors Porcelain Company	26	55-gal drums
1965	U.S. Geological Survey and Denver Research Institute	1	55-gal drum
1966	U.S. Geological Survey and Denver Research Institute	3	55-gal drums
		1	30-gal drum
		1	carton
1967	U.S. Geological Survey and Denver Research Institute	2	55-gal drums
	U.S. Geological Survey	1	55-gal drum
	U.S. Department of the Interior	2	55-gal drums
1968	Dow Construction, U.S. Geological Survey, Department of the Interior, and VA Hospital	8	55-gal drums
		1	30-gal drum
		2	wooden boxes
1970	TOSCO	25	55-gal drums
	VA Hospital	1	55-gal drum
	U.S. Geological Survey	16	55-gal drums
	Coors Porcelain Company	44	55-gal drums
1971	U.S. Geological Survey	21	55-gal drums
	Denver Research Institute	5	55-gal drums
	U.S. Fish and Wildlife Commission	13	55-gal drums

a. Solar pond solids shipped as evaporator salts (Series 745 sludge).

4.4.16 Waste Quality Program

The waste quality program progressed from very little control over the quality of the waste shipped in the 1950s to a fully instituted quality program in the 1970s, continuing until the termination of shipment receipt at INL in 1989.

The first quality action was taken after the first waste shipment to INL in April 1954. A letter from G. V. Beard, Chief of the Health and Safety Branch, Idaho Operations Office, to John Epp, Assistant Director, Chemical Laboratories, Dow Chemical Company, RFP, dated May 5, 1954 (see Appendix A) described liquid leakage from seven drums. The letter requested action to preclude free liquids in future shipments. This letter initiated a quality effort to eliminate free liquids and to provide for absorbing any liquids that might develop during transport to INL.

Continued correspondence between INL and RFP initiated improvements and upgrades in waste packaging through drum and crate liners, closure mechanisms, and segregation of contents.

In October 1968, the U.S. Department of Transportation (DOT) issued new regulations (hazardous materials regulations [49 CFR 171–180]) for shipping radioactive materials that were in substantial conformance with the 1967 regulations of the International Atomic Energy Agency. Shipping methods, packaging procedures, and shipping containers were upgraded to meet these new regulations. Packaging line-generated waste required using drums that met specifications of the Interstate Commerce Commission, such as the 17H and 17C drums. The drum changeover was not new to RFP as the 74 series of sludge changed from 17H to 17C drums to take advantage of the maximum weight limit from 300 kg (660 lb)/drum to 400 kg (880 lb)/drum. This changeover was instituted in 1966. The upgraded packaging was certified by AEC Albuquerque Operations Office (see Appendix C).

In the late 1960s, installing drum counters for waste assays of SNM required a more definitive matrix to apply matrix density correction factors. Consequently, the five waste types that describe the waste shipped previously were replaced by item description codes for specific materials such as graphite, fire brick, raschig rings, sand, slag, and crucibles.

In 1970, the AEC directive (AEC 1970) on the disposal of TRU waste forced RFP to establish a formal quality assurance program for waste material. This quality assurance program instituted quality control and inspections on waste containers, packaging materials and procedures, personnel training certification, improved labeling, upgraded waste descriptions, and a demand for methods of NDA. The upgrades and improvements in waste packaging at RFP in the 1970s are discussed and described by Wickland (1977).

Since this report addresses mainly pre-1970 RFP waste, continuing quality improvements that occurred during the 1970s and 1980s will not be further discussed. In general, quality and control of radioactive waste shipped to INL improved gradually from 1954 to 1969. Waste containers, packaging materials, and procedures were standardized; other improvements include descriptive labeling and a more precise description of waste categories.

5. DEVELOPMENT OF WASTE CONTAINER STANDARDIZATION AND IDENTIFICATION

The shipping containers employed to ship radioactive waste to INL evolved from any available and suitable container to standardized containers that were quality controlled and performance tested. The upgrading and improvements instituted were driven by AEC and DOE directives, DOT and Interstate Commerce Commission regulations, and INL criteria for receiving waste.

5.1 Generator Identification

From 1954 through 1969, the containers generated by RFP were identified by a prefix representing the building generator number followed by a serial number usually assigned by the building generator and coordinated by the Waste Coordination Group. These prefixes are shown in Table 8 for pre-1970 waste shipments.

In 1970 and thereafter, radioactive waste was characterized as TRU waste and non-TRU waste. Table 9 lists the sources of TRU and non-TRU waste by prefix numbers and buildings.

Table 8. Identification of waste container generator pre-1970.

Prefix Number	Building Number	Building Mission
Plutonium Waste		
122	122	Medical treatment
123	123	Health physics laboratory
59	559	Plutonium analytical laboratory
71	771	Plutonium recovery and recycle
71(596)	771, 776, 777	1969 fire waste
741	774	First-stage sludge
742	774	Second-stage sludge
743	774	Grease Plant (organic) sludge
744	774	Cemented liquid waste
745	774	Evaporator salts
746	774	Empty contaminated drums
776	776	Plutonium manufacturing
77	777	Plutonium component assembly
78	778	Plutonium laundry
79	779	Research and development laboratories
79A	779A	Research and development laboratories
81	881	HEU and plutonium waste ^a
91	991	Plutonium and HEU component assembly
95	995	Sewage treatment plant

Table 8. (continued).

Prefix Number	Building Number	Building Mission
Highly Enriched Uranium Waste		
22	122	Medical treatment
23	123	Health physics laboratory
81	881	HEU fabrication, chemical recycle ^b
83	883	HEU forming
86	886	HEU criticality exposure assembly
89	889	HEU decontamination facility
91	991	HEU component assembly
Depleted Uranium Waste		
31	331	Temporary development facility
41	441	DU analytical laboratory
44	444	DU fabrication
47	447	Roaster oxide
83	883	DU forming
Beryllium Waste		
41	441	Beryllium analyses
44	444	Beryllium fabrication
47	447	Beryllium fabrication
71	771	Beryllium component destruction
741	774	Beryllium in first-stage sludge
742	774	Beryllium in second-stage sludge
745	774	Beryllium in evaporator salts
76	776	Beryllium components handled
77	777	Beryllium components handled
79A	779A	Beryllium components processed
83	883	Beryllium forming/descaling
a. Processing of returns b. HEU cleanout started in 1965. DU = depleted uranium HEU = highly enriched uranium		

Table 9. Post-1970 transuranic and nontransuranic waste generators.

Prefix Number	Building Number	Building Mission
Transuranic Waste Generators		
59	559	Plutonium analyses
07	707	Plutonium fabrication
71	771	Plutonium recovery and recycle
71(596)	771	1969 fire waste
741	774	First-stage sludge
742	774	Second-stage sludge
743	774	Grease Plant (organic) sludge
744	774	Cemented liquid waste
745	774	Evaporator salts
746	774	Empty contaminated drums
76	776	Plutonium manufacturing
77	777	Plutonium component assembly
78	778	Plutonium laundry
79	779	Research and development laboratories
79A	779A	Research and development laboratories
81	881	HEU and plutonium waste ^a
95	995	Sewage treatment plant
Nontransuranic Waste Generators		
23	123	Health physics laboratory
31	331	Temporary development facility
44	444	Depleted uranium fabrication
47	447	Roaster oxide
865	865	Research and development facility
81	881	HEU cleanup and decommissioning
83	883	Depleted uranium forming
86	886	HEU criticality exposure
89	889	HEU decontamination facility

a. Processing of returns.
HEU = highly enriched uranium

Two special projects carried out in Building 881 generated TRU waste besides the processing of returns. The first used U-233, and the second used Np-237. These two projects were not routine operations and were of short duration.

5.1.1 Post-1970 Identification of Containers

Using drum prefix numbers to identify building generators was discontinued in 1971. A new system was installed using prefix numbers from 01–99 to identify the material balance area that generated and packaged the waste container. An example is illustrated below.

02- 000001
↑ ↑
Prefix Container
material serial
balance area- number
identification

A container serial number was assigned by the department responsible for the material balance area; however, Building 774 sludge still used its original prefix numbers because Building 774 did not have any material balance areas. This system was used only for TRU waste.

When Rockwell International became the contractor in 1975 for RFP, the waste container identification system was altered again. White-painted drums were used for TRU waste and were serialized by the warehouse. All white drums were issued with a metal tag with the drum serial number provided by the warehouse. The material balance area and department container serialization were retained as indicated below.

Container serial number
↓
DXXXXX-02-000001
↑ ↑
Drum serial Prefix
number material
balance area-
identification

This container identification system, started in 1975, has continued to the present.

5.2 Waste Containers

The majority of waste containers shipped to INL were 55-gal drums. The second-place drum was the 30-gal drum. A small number of 40- and 45-gal drums was shipped in the 1958-1961 timeframe. A very limited number of 20-gal drums also was shipped in the 1958-1961 timeframe. In 1972, an 83-gal drum was introduced for Building 774 sludge and other plutonium-bearing waste. However, the use of 83-gal drums was discontinued in 1973. Occasionally, drums of a different capacity were received from offsite and were shipped to INL as indicated in Table 7.

The original source of drums was a mixture of vendor product drums (i.e., drums that had contained solvents or other materials and were reused to dispose of waste) and newly purchased drums. Building 774 started to use new drums for sludge in April 1958. The gradual buildup of waste from the increased plutonium manufacturing mission in the late 1950s and early 1960s increased the need to purchase new drums.

Initially, 17H drums were used but proved unsatisfactory for packaging Building 774 sludge. The 17H 55-gal drum had a weight limit of 299.4 kg (660 lb), which limited the packaging of 774 sludge. In the 1965–1966 timeframe, 17C 55-gal drums were purchased for sludge as the weight limit was 399.2 kg (880 lb) per drum. For consistency onsite, the 17C drum became the standard drum.

Shipping waste in wooden boxes began in December 1954. Wooden boxes accommodated items that were too large or too heavy for drum packaging. Originally, wooden box dimensions were tailored to fit the item to be shipped. As the use of wooden boxes (crates) increased, the box size was standardized to 4 × 4 × 7 ft to facilitate loading into trailers and railcars. At the packer's discretion, some items were wrapped in plastic before being placed in the box, such as items that were bulky, had sharp edges, had surface contamination, or had come loose or become unwrapped during shipping. Coating plywood boxes with fiberglass began in July 1971.

Other forms of waste shipped during the late 1950s were several tanks, a couple of small metal boxes, and two cylinders. Cardboard cartons were occasionally used for special items; however, in 1967, cartons were used to ship unleached spent HEPA filters. Shipment of CWS filters in drums or cartons began in March 1955.

5.3 Waste Packaging

Packaging radioactive waste evolved from a simple liner system to a maximum containment configuration. The packaging scheme was governed by the type of waste to be shipped, packaging and shipping regulations, and the receiving site's disposal criteria in effect at that time.

5.3.1 Non-Line-Generated Waste

Non-line-generated waste originated outside the glove-box lines but within a plutonium handling facility. Other sources were from the uranium processing buildings. Any sharp or cutting edges of waste were taped to prevent punctures. Waste was placed directly into a drum or wooden crate, which had a polyethylene (5- or 8-mil) liner. The liner was taped closed, closed by heat sealing, or both.

5.3.2 Line-Generated Waste

Items capable of puncturing the drum liner were taped or placed in 8-mil fiberboard sleeves with a polyethylene wrapping. Powdery items and small solid items were sealed within paint cans, plastic bottles, or other similar containers. The contaminated material was removed from the glove-box line through a bag-out procedure. The sealed polyethylene bag was placed in a 17H or 17C drum that had a 5- or 8-mil polyethylene liner. When full, the liner was taped closed, and the drum lid was secured to the drum body by a 12-gauge, bolted-ring closure system.

5.3.3 Crated Waste

Large bulky items, such as metal scrap, light fixtures, tool machines, lumber, piping, hoods, or air ducts, that could not fit in 55-gal drums were placed in wooden crates. These objects were contaminated externally to varying levels with plutonium, and in some cases with HEU. The crate was lined with an 8-mil polyethylene sheet. Heavy items were secured by bolting to the crate skids. When full, the liner was taped or heat sealed, and the lid was nailed to the crate body. The closed crate was banded with 1.25-in.-wide steel straps in at least four positions.

5.3.4 Series 774 Sludge Waste

The majority of sludge waste was generated by the Liquid Waste Treatment Plant (Building 774). Dry Portland cement was placed into a 17C or 17H 55-gal drum lined with a 5- or 8-mil polyethylene liner. Additional dry Portland cement was interspersed with the sludge during the drum-filling cycle. After completing the filling cycle, the liner was taped closed, and a second quantity of dry Portland cement was placed on top of the liner. The drum was sealed with a 12-gauge bolted ring.

5.3.5 Series 743 Grease Sludge

The organic-based (grease) sludge was produced mainly by the Liquid Waste Treatment Plant in Building 774. An oil-dry absorbent was placed in a 17C or 17H 55-gal drum lined with a 5- or 8-mil polyethylene liner. The greaselike sludge was added to the lined drum. Microcel, manufactured by Johns-Manville, was used as a hardener for the sludge. After filling, the liner was taped closed, and hardener was placed between the liner and drum lid. The lid was sealed with a 12-gauge bolted ring.

5.3.6 Neutralized Hydrochloric Acid Solutions

Waste hydrochloric acid solutions were neutralized with sodium hydroxide and solidified with magnesia cement within the drum. Lining the drum was 8-mil polyethylene. After sealing the liner with tape, magnesia cement was placed between the sealed liner and the drum lid. The lid was sealed with a 12-gauge bolted ring.

While polyethylene bag-out bags were preferred for incineration, PVC bag-out bags also were used. Waste generators were encouraged to use PVC bags when waste was not to be incinerated.

5.3.7 Post-1970 Packing

The packaging employed for the 1970–1972 timeframe for line-generated waste consisted of one or two bag-type polyethylene liners depending on the type of waste being packaged. The individual packages placed within the drum were polyethylene or PVC bags taped closed. Cardboard liners placed inside the inner bag liner were used for abrasive waste such as graphite molds or fire brick.

In the latter part of 1972, use of the 90-mil, rigid polyethylene liner began. The rigid liner was placed inside the drum; one or two polyethylene drum liner bags were placed inside the rigid liner. The polyethylene bags were taped closed, and the top of the rigid liner sealed. From 1972 to early 1982, Oil-Dri absorbent was added (0.9–1.9 L [1–2 qt]) to the top of the sealed outer drum liner. Vermiculite was substituted for Oil-Dri after February 1982. A more complete packaging description for post-1970 waste can be located in Clements (1982).

5.4 Packaging and Container Codes

Copies of load lists for trailers and atomic materials rail transfer (ATMX) railcars for the 1950s and 1960s were forwarded to INL personnel to reconcile shipper-receiver records. A variety of codes was employed on the load lists to indicate the packaged contents and the containers involved. The content-code Types I through V have been described previously in Section 4, “Waste Characterization.”

The Bureau of Explosives issued permits to RFP covering hazardous material containers for shipping. The Bureau of Explosives codes that appeared frequently on the load lists are shown in Table 10.

Table 10. Bureau of Explosives codes on the load lists.

Bureau of Explosives Permit Number	Use
2056	Plywood boxes containing machinery and glove boxes
2057	Second-hand 55-gal drums containing trash and dry waste
2058	16-gauge Interstate Commerce Commission Spec. 17C drums containing sludge
2059	Interstate Commerce Commission 12B cartons containing CWS filters
2060	Plywood boxes containing CWS filters

CWS = Chemical Warfare Service

Occasionally, the symbol “MTD” was indicated on the load lists, which indicated empty drum(s).

The label notation “Red” or “Blue” on the load lists indicated plutonium content. Containers with less than 15 g of plutonium were labeled in blue; those with 15 g or more were labeled in red.

The notation “LLD” for a given drum indicated the drum was lined with lead. Lead drum liners were used to ensure the external radiation requirement was met.

Load lists in the early 1970s used the notation “nret,” which stood for nonretrievable (i.e., not TRU) waste. Waste identified as nret was sent to INL for burial.

The designation of “Plant Waste” meant that the waste was generated outside of any process or support facility.

The labeling of radioactive containers was based on transportation and packaging regulations in effect at the time of shipment to INL; however, exemptions were granted for a few shipments, which could not meet the standard regulations.

6. WASTE ASSAY

At startup of RFP, a waste policy was in place to declare the loss of SNM through the concept of normal operating loss. The waste generator was responsible for assigning an SNM value to the waste generated. For liquid waste, a chemical or a radiometric assay for SNM was performed. Initially, an estimating procedure was used for solid waste based mainly on a “by-difference” approach coupled with operating experience.

These assay methods proved to be inadequate as the “material unaccounted for” grew to an unacceptable level. Zodtner and Rogers (1964) stressed the need for improved methods of assaying plutonium in the waste streams and identified several issues that contributed to material unaccounted for. These issues are listed below and are associated mainly with assignments to normal operating loss:

- Understatement of plutonium in graphite waste
- Understatement of plutonium on HEPA filter waste
- No available plutonium assay for in situ combustible and other solid waste forms (no representative sample available)
- Inadequate liquid assay methods.

6.1 Liquid Waste Assay

The majority of plutonium-contaminated aqueous waste was generated by the plutonium recovery facility, Building 771. See Table 11 for types of solutions transferred from Building 771 to the Liquid Waste Treatment Plant for the month of December 1961 and for aqueous solutions received from other buildings.

Determining plutonium and americium in liquid waste solutions was accomplished by radiometric and chemical titration methods. The radiometric method often required large dilutions of the original solution, which added to the inaccuracy of the assay. Obtaining a representative sample from raschig ring tanks also was difficult. Measuring liquid volume accurately within a tank was another issue. A tank calibration crew was organized to solve the tank calibration issue. A group also was formed to prepare known plutonium solutions coupled with statistical assistance to develop precise and accurate data.

Waste solutions originating from Building 771 were collected in critically safe tanks awaiting transfer to Building 774. The tanks were assayed for plutonium and Am-241 content. If below discard limits for plutonium, the solutions were transferred to Building 774 for disposal treatment. The plutonium content of the solutions was taken as a normal operating loss. On a batch basis, plutonium content was pro-rated per drum of first-stage sludge produced. The plutonium content transferred to the second-stage sludge was pro-rated also. The HEU processed into second-stage sludge was pro-rated also and taken as a normal operating loss when received by Building 774.

Because of concern for improved criticality control and for upgrading the assay, a second set of critically safe tanks was installed in the early 1970s. The sampling program was improved by having both the generator and the Liquid Waste Treatment Plant sample the same tanks. The results had to agree statistically before the second set of tanks could be transferred to Building 774. Improved analytical methods also were developed.

Table 11. Liquid waste solutions received for treatment December 1961.

First-Stage Treatment		
Source	Description	Quantity (gal)
Building 771	Ion column effluent	1,656
	Raffinate	5,077
	Cooling waste	19,800
	Nash pumps (vacuum)	4,653
	Distillate	3,750
	Miscellaneous	423
Total		35,359
Second-Stage Treatment		
441	Depleted uranium analyses	14,200
444	Depleted uranium and beryllium manufacturing	34,800
771	From first stage	39,950
771	Laundry	30,650
776	Plutonium manufacturing	13,800
881	Highly enriched uranium manufacturing	76,700
Total		210,100

6.2 Raschig Ring Tanks

The concept of employing raschig rings for nuclear criticality control was extended in the early 1960s to plutonium tanks. Raschig rings were fabricated from boron silicate glass as a hollow cylinder with a 0.72-in. outside diameter, 1.75-in. length, and wall thickness of 0.25 in. Boron acts as a neutron absorber, thereby providing a degree of criticality control for tanks holding plutonium-bearing solutions. Building 771 waste tanks were raschig ring tanks.

Several process problems were associated with raschig ring tanks. The first was the buildup of plutonium sludge on the raschig rings, thereby requiring the rings' removal and replacement with new rings. The second was the difficulty of achieving solution homogeneity within the tank. The third was ring breakage during air sparging to accomplish solution homogeneity. The fourth was thinning of the rings from fluoride ion attack. Fluoride ions were used to facilitate plutonium oxide dissolution. Calcium fluoride slag reprocessing for plutonium recovery also provided a source of fluoride ions. Ring thinning, breakage, and accumulated sludge buildup adversely affected tank calibration.

Studies of solution circulation favored air sparging over circulation from bottom to top of the tank; however, continued studies demonstrated the inconsistencies of air sparging when applied to different tanks. A lengthy air sparging cycle was necessary to obtain proper solution mixing. This problem also contributed to the requirement for two analyses of a tank before shipping waste solutions to Building 774.

The waste solution mixing procedure was a lingering nuclear accountability concern because of doubts about analytical sample representation.

Raschig ring tanks were favored for some plutonium solutions because they required less floor space than geometrically safe tanks; however, annular tanks gradually were replacing raschig ring tanks when floor space permitted the changeover. This was not the case for waste solution tanks. Raschig rings that had been removed were leached with nitric acid to remove the residual sludge, then washed in water, dried, and packaged into 55-gal drums for shipment to INL.

6.3 Solid Waste Assay

During the 1950s and early 1960s, chemical assay and radiometric analysis were the two analytical methods for SNM determinations. X-ray fluorescence methods also were developed for plutonium analyses in the early 1960s but were used mainly on production samples.

Zodtner and Rogers (1964), in addressing issues of material unaccounted for, called for developing procedures for NDA of solid waste. Consequently, an R&D effort was launched in the mid-1960s to develop gamma-neutron counting systems applicable to solid waste packaged in drums. The initial experimental R&D drum counter was activated in 1964 and was located in Building 771. The first production drum counter was installed in Building 771 in 1969.

Continuing R&D activities and electronic advancements produced sophisticated counting systems that provided helix scanning, segmented gamma scanning, low-resolution gamma assaying, alpha-neutron corrections, background corrections, matrix density adjustments, and computerization of radiometric calculations. Systems were installed to assay small containers, drums, crates, and HEPA filters. By the late 1970s and early 1980s, systems had been developed and were operating to assay all item description codes. However, these advanced systems were not available for assaying the majority of pre-1970 waste sent to INL. See Appendix D for reports and references describing these systems and their operation.

6.4 Production Nondestructive Assay Systems

In 1984, the “Handbook of the Rocky Flats Plant Production Non-Destructive Assay Systems” was compiled by Bill Ulbricht (see Appendix E). The handbook described the counting systems and the container and applicability of item description codes and listed the building location for each counting system and its operational status. In 1987, the handbook was reissued to upgrade the information on NDA (see Appendix F). These two handbooks provide an insight into the state and applicability of the NDA systems used during the 1970s and 1980s for assaying solid waste.

6.5 Plutonium Waste Discards

In 1964, the first experimental NDA system for drum counting was installed. This counting system assayed for plutonium content in waste drums shipped to INL. The advancements and improvements realized in the second-generation NDA drum counting systems and the development of correction factors for matrix density, alpha-neutron reactions, and electronic anomalies prompted a review of the data for understated plutonium assays from the 1964 counting system. Consequently, a review—covering from February 1968 to June 1971—was undertaken of the counting data for 24,000 drums assayed with the 1964 drum counting system.

Bidwell, Chanda, and Cartwright (1973) address material unaccounted for in the INL drum field, and according to Table 11 in the report, total plutonium weight was understated by about 17.6 kg (38.8 lb)

for 15,795 drums at INL (see Appendix G). These drums suspected of being understated were reviewed based on their gamma and neutron counts recorded by the 1964 drum counter. Based on new correction factors, operating experience, and recounts of drums with similar item description codes in the RFP backlog drum field, a plutonium estimate was derived for the suspect drums at INL. These plutonium estimates are summarized in Table 12 for individual item description codes. Two of the INL suspect drums were assayed at the National Reactor Testing Station. The comparison of assays is shown in Table 13.

Table 12. Suspect drums shipped to Idaho National Laboratory.

Number of Drums	Item Description Code	Description	Original Rocky Flats Plant Plutonium Weight (g)	Estimated Plutonium Weight (g)
1	300	Graphite molds	0	7
1	320	Heavy non-special-source metal (such as tantalum, tungsten, and platinum)	5	9
8	330	Dry combustibles (paper and rags)	25	2,754
1	336	Wet combustibles (paper and rags)	0	27
3	338	Filter media	49	92
1	372	Grit	45	65
2	440	Glass (except raschig rings)	1	18
3	441	Unleached raschig rings	21	1,486
33	480	Light non-special-source metal (such as aluminum, copper, and beryllium)	268	1,208
53	All	Totals	414	5,666
		Difference	5,666-414	5,252

Table 13. Suspect drum comparison.

Drum Number	Item Description Code	National Reactor Testing Station Plutonium Weight (g)	Original Rocky Flats Plant Plutonium Weight (g)	Estimated Rocky Flats Plant Plutonium Weight (g)
57493	330	500	0	476
59682	480	300	0	319

Plutonium discards to waste (i.e., normal operating losses) were tabulated by D. L. Ziegler for calendar year 1967. The total plutonium discard written off as shipped to INL for Calendar Year 1967 was 70,382 g (155.2 lb). Ziegler (see Appendix H) provides tables for Calendar Year 1967 waste covering six groups of waste listed below:

- Liquid waste from the plutonium recovery area
- Building 774 waste
- CCl_4 —oil converted to grease in Building 774
- Non-line-generated hot waste
- Line-generated waste
- Crated waste.

6.6 Economic Discard Limits

6.6.1 Background

Recognition in the late 1950s and early 1960s that significant quantities of plutonium were being lost through the solid waste streams emphasized the need for plutonium assay systems tailored to assay containers of waste. Also needed were plutonium recovery systems designed to process solid and liquid waste streams.

In 1960, a project was initiated to provide the necessary plutonium recovery capability and capacity for treating low-level solid residues and liquid waste solutions. This project included the development of an NDA drum counter. Construction was completed in 1962, and the additional recovery systems were started in the mid-1960s. The first workable drum counter was installed in Building 771 in 1964. This experimental drum counter provided the necessary data and operational experience for continuing upgrades to establish a full production drum counter in 1969.

6.6.2 Establishing Economic Discard Limits

Before establishing economic discard limits, the decision to process a given residue drum for plutonium recovery was made arbitrarily by operations supervision. This inconsistent procedure showed that a better method was needed to determine the feasibility of recovery processing. In addition, the processing of all waste was not economically necessary.

The RFP approach was to establish a system of economic discard limits. As provided by the AEC and later DOE, the cost of plutonium recovered and processed to a pure metal state was compared with the value of producing new reactor weapons-grade plutonium metal. The break-even point was the economic discard limit for a given residue. These limits were ordinarily expressed in (1) gram of plutonium per gram of residue for solids and (2) gram of plutonium per liter of solution for liquids. Any residue item above the discard limit required processing, and any residue item below the discard limit was considered waste.

6.6.3 Calculating Economic Discard Limits

The economic discard limits were calculated using Equation (1):

$$D = \frac{(L)(T_{(S)})}{(C - F)(E)} \quad \text{or} \quad D = \frac{(L)(T_{(L)})}{(C - F)(E)} \quad (1)$$

The symbols of the equation are explained below.

Symbol	Definition	Units
D =	Economic discard limit concentration	kgPu/1,000 L or kgPu/kg residue
L =	Labor cost per productive man hour	\$/hour
T _(L) =	Time required to process one kiloliter of the original liquid residue through residue recovery	hour/1,000 L
T _(S) =	Time required to process 1 kg of the original solid residue through residue recovery	hour/kg
E =	Total process efficiency, fraction of plutonium in residue that is converted to metal	kgPu/kgPu
C =	DOE cost to produce new weapons grade plutonium	\$/kgPu
F =	Rocky Flats Plant cost to produce plutonium metal from concentrated plutonium nitrate feed	\$/kgPu

To determine these limits, data were collected on material accountability; processing rates and efficiency; and labor, material, and support costs. These data were collected to make the initial determination and also on a continuing basis. All plutonium-bearing residues were divided into categories with similar processing requirements. Each such category was assigned a digital item description code for tracking purposes. Similarly, each finite step of residue recovery processing was assigned an operations code number. All such costs as direct labor cost, supplies and materials, and maintenance conducted on that step were charged to that assigned code number.

For each period between inventories (usually 1 month), the gross plutonium weights of residue material charged to the operation, the net weight recovered, and the total cost of the operation were determined. The cost of processing per unit weight of residue processed, as well as the cost per gram of plutonium handled through the operations step, was calculated mathematically. The total cost per gram of plutonium was the sum of all operational steps required to convert the plutonium to metal.

Because unit recovery costs could vary significantly from month to month, for instance as a result of major maintenance work, economic discard limits were normally calculated and revised annually. In later years, when the generation rate of a particular category of residue exceeded processing capacity, a factor was added to allow for the construction of new facilities, amortized over a 10-year period.

As indicated above, economic discard limits and item description codes were coupled and driven by improved NDA procedures and recovery systems. Additional description codes and corresponding economic discard limits were added as segregation requirements became known for obtaining more residue homogeneity to achieve more accurate NDA results.

Anderson, Putzier, and Ziegler reported the discard limits for Fiscal Year 1969 (shown in Table 14) in their internal report reproduced in Appendix I.

Introduction of economic discard limits probably began with drum counting capability, which has been placed in the 1966–1967 timeframe. A letter from William F. Romine, RFP Traffic Manager, dated December 23, 1968 (see Appendix X), defines line-generated waste coupled with an economic discard limit range as: “Line-generated wastes are graphite molds, filters sludge, insulation, glass, washables, combustibles, metals, and miscellaneous residues with plutonium discard limits ranging from 7×10^{-3} g/g to 3.0×10^{-4} g/g.” This letter also indicates that economic discard limits were in place in 1968.

Table 14. Economic discard limits—Fiscal Year 1969.

Category	Discard Limit (gPu/g Total)
Sweepings	0.007
Sludge	0.007
Magnesium oxide sand	0.007
Ion exchange resin	0.007
Incinerator ash	0.007
Sweepings heels	0.007
Ash heels	0.007
Glass and ceramics	0.0005
Scarfed molds	0.00035
Graphite flow residue	0.002
Chemical Warfare Service filter $2 \times 2 \times 1$ ft	24.0 g/filter
Dry box filters $8 \times 8 \times 4$ in.	3.0 g/filter
Washables	0.0006
Combustibles	0.0007
Miscellaneous scrap metal	0.0003
(see Appendix I)	

7. WASTE TRANSPORTATION

The RFP waste was transported to INL using tractor trailers and ATMX railcars. A combination of trailers on flatbed railcars also was employed.

7.1 Trailers

Several commercial trucking firms were contracted by RFP Traffic Group to haul waste drums and crates (boxes) from RFP to INL. The cargo consisted mainly of 55- and 30-gal drums with an occasional drum of another capacity. Crates (boxes) were the second type of container hauled. Cardboard containers also were used for CWS and HEPA filters.

Maximum drum capacity per trailer was 164, but trailer loads usually were less with a range of 62 to 154 drums per trailer. The total load weight was a governing factor.

The first shipment of drums to INL in April 1954 leaked a small amount of liquid that contaminated the trailer floor. The AEC Idaho Operations Office personnel recommended using an absorbent floor covering to absorb any leaking liquid, but this was discontinued when the policy of not putting liquids in drums was implemented.

7.2 Atomic Materials Rail Transfer Railcars

Higher levels of plutonium waste required a Type B package to meet AEC (1973) and DOT regulations, which were to take effect in the late 1960s. Rocky Flats Plant generated about 3,000 55-gal drums and 36,000 ft³ of crates having a plutonium content requiring Type B packaging. The existing containers and those under development were either inadequate or too expensive to accommodate the waste shipments to INL.

The 600 series ATMX railcar designed by Sandia Laboratory for hauling explosive materials was selected for certification as a Type B container. The ATMX-600 railcar was certified, and two were assigned to RFP by the AEC. Eventually, six railcars were assigned to haul RFP waste to INL.

The ATMX-600 railcar could accommodate 216 55-gal drums loaded individually and a significant number of crates depending on their weight. The ATMX-600 railcar also could accept two cargo carriers, which reduced the loading time significantly but reduced the total drum load to 132 per railcar. ATMX-600 railcar shipments to INL commenced in 1969 and continued until 1989.

A more thorough description of the ATMX-600 railcar is provided by Adcock (1970). The safety analysis report describing packaging for the ATMX-600 railcar was issued by Adcock and McCarthy (1974).

7.3 Piggy-Back Trailers

A special request to ship plutonium-contaminated residues to INL that had been stored at RFP was granted by AEC Albuquerque Operations Office in 1963 because RFP did not have the capacity then to process this residue backlog. Trailers containing the waste were “piggy-backed” on flatbed railcars and escorted by health physics personnel for the journey to INL. This arrangement continued until all the residue drums were shipped in 1964.

7.4 Shipping Forms

In the 1950s and 1960s, RFP began using the AEC Idaho Operations Office shipping form, “Solid Radioactive Waste Disposal Order and Shipping Data.” The form consisted of the following three sections:

- Section I identifies shipper and receiver
- Section II describes waste
- Section III verifies disposal.

Sections I and II were completed by the shipper (RFP), while Section III was completed by receiving (INL) personnel. Figure 1 is a copy of a completed form. Shipments were identified by two systems: (1) Dow serial number and (2) Rocky Flats seal number. As shown in Figure 1, the Dow serial number was 66-57-B with a Rocky Flats seal number of RF-3790. In the latter part of the 1960s, health physics numbers were used instead of Dow serial numbers. A more sophisticated radioactive waste form (ID-135) was employed in the 1970s by INL. Figure 2 shows the first page of Form ID-135.

To record trailer and ATMX-600 railcar loadings, RFP used a load-list form. The load list was completed by personnel loading the waste. Consequently, the load lists recorded for pre-1970 waste were handwritten. Figure 3 is a copy of a load list. The load lists in the late 1960s became known as waste disposal sheets. In the 1970s and 1980s, the load lists were computerized and were organized to provide data for completing Form ID-135. Figures 4 and 5 are copies of the first two pages, respectively, of a computerized load list.

FORM ID-110A (8-65)	U. S. ATOMIC ENERGY COMMISSION IDAHO OPERATIONS OFFICE SOLID RADIOACTIVE WASTE DISPOSAL ORDER AND SHIPMENT DATA	REFERENCE: IDM 6506-7 TERMS: SEE REVERSE SIDE OF FORM
RPDOW SRI 07/08/66 1128 2 3 DISPOSAL LOCATION: PIT 4		
SECTION I		ORDER NO. <u>65051 G</u>
TO: U. S. ATOMIC ENERGY COMMISSION IDAHO OPERATIONS OFFICE IDAHO FALLS, IDAHO 83401		FROM (NAME & ADDRESS OF AGENCY) The Dow Chemical Company Rocky Flats Division Post Office Box 888 Golden, Colorado 80401
ATTENTION: <u>R. L. Hayden</u>		BILL TO: (IF DIFFERENT THAN ABOVE)
THE COMMISSION IS HEREBY REQUESTED TO DISPOSE OF THE RADIOACTIVE WASTE DESCRIBED ON THIS ORDER ACCORDING TO THE TERMS AND CONDITIONS SPECIFIED HEREON.		
ORIGINATOR <u>E. A. [Signature]</u> (SIGNATURE)	WD Coordinator <u>[Signature]</u> (TITLE)	<u>July 8, 1966</u> (DATE)
ACCEPTED FOR THE ATOMIC ENERGY COMMISSION BY:		
<u>P. E. [Signature]</u> (SIGNATURE)	CFP HP Supervisor <u>[Signature]</u> (TITLE)	<u>7/21/66</u> (DATE)
SECTION II DESCRIPTION OF WASTE		
Trlr. No. 800203 Dow S.N. 66-57-B Seal No. RP-3790		
1. TOTAL NO. OF PACKAGES	<u>17</u>	VOLUME IN FT. ³ <u>2,074</u> WEIGHT <u>25,200</u>
2. COMPLETE DESCRIPTION OF CONTENTS AND PACKAGING		
<u>17 Wdn boxes of scrap metal, equipment, filter, etc.</u>		
3. CLASSIFICATION: SECRET <u>CONFIDENTIAL</u> CATEGORY <u>Unclassified</u>		
US MATERIAL TYPE <u>1</u> AMOUNT <u>NET</u> ISOTOPE <u>3</u>		
4. PROPOSED MEANS OF TRANSPORTATION <u>D&RG & UP railroad</u> SHIPPING DATE <u>7-11-66</u>		
5. RADIOISOTOPES CONTAINED <u>plutonium and uranium</u>		
6. MAXIMUM RADIATION AT SURFACE OF <u>Trailer</u> <u>2.0 mr/hr</u> TOTAL CURIES <u>not feasible</u>		
7. ASSOCIATED HAZARDS: <u>None</u>		
* These amounts as of <u>the end of the calendar year</u>		
SECTION III (TO BE COMPLETED BY PERSON WITNESSING DISPOSAL)		
DISPOSAL WAS MADE BY MEANS OF <u>Burial in Pit # 4</u>		
AT <u>190' West of the S/E Monument</u>	ON <u>7-15-66</u>	
(LOCATION)	(DATE)	
<u>M.W. Breshear</u>	<u>[Signature]</u>	<u>7-15-66</u>
(SIGNATURE)	(SIGNATURE)	(DATE)

Figure 1. Form ID-110A, "Solid Radioactive Waste Disposal Order and Shipment Data."

File

FORM ID-135
(Rev. 6-77)

ENERGY RESEARCH & DEVELOPMENT ADMINISTRATION
IDAHO OPERATIONS OFFICE

RADIOACTIVE WASTE FORM

REFERENCE
IDW 9511

GFL 91-5480 SN7802 5 *deal Wm-10289*

DATE: MONTH DAY YEAR
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31

TO BE ON ALL COPIES

SECTION 0

DATE: MONTH DAY YEAR
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31

TO BE ON ALL COPIES

SECTION 1

DESCRIPTION OF WASTE

TRAN 780205 1.008 + 0.35 3508 + 0.42 2408 + 0.44

SECTION 2

DESCRIPTION OF WASTE

8xw 9 112 FBLdg-774-EVAPORATOR-SALT

SECTION 3

DESCRIPTION OF WASTE

DATE SHIPPED 02-10-78

GRAMS 0.00057 g PH

Figure 2. Form ID-135, "Radioactive Waste Form."

WASTE DISPOSAL

No.	Drum No.	Gross Weight	Drum Size	Type	No.	Drum No.	Gross Weight	Drum Size	Type
1.	✓ 71-437	1,600	84x52x18	V	32.	✓ 71-616	1,410	84x52x18	V
2.	✓ 71-444	1,600	84x52x18	V	33.	✓ 71-492	1,200	84x52x18	V
3.	✓ 71-483	1,700	84x52x18	V	34.	✓ 71-477	1,000	84x52x18	V
4.	✓ 71-484	1,500	84x52x18	V	35.	✓ 71-493	1,100	84x52x18	V
5.	✓ 71-485	1,700	84x52x18	V	36.	✓ 71-498	1,200	84x52x18	V
6.	✓ 71-467	1,600	84x52x18	V	37.	✓ 71-492	900	84x52x18	V
7.	✓ 71-422	2,100	84x52x18	V	38.	✓ 71-134	1,800	84x52x18	V
8.	✓ 71-622	1,800	84x52x18	V	39.	✓ 81-623	1,900	84x52x18	V
9.	✓ 94-95	1,200	84x52x18	V	40.	8	10,300		
10.					41.				
11.	9	14,900			42.				
12.					43.				
13.					44.				
14.					45.				
15.					46.	40 of 8	2060		
16.	9	14,900			47.	130 of 8	2056		
17.	8	10,300			48.				
18.					49.				
19.	17	25,200	2.074	13	50.				
20.					51.				
21.					52.				
22.					53.	44 of 8 type V	2-WB	2,100	244 ft
23.					54.	71 of 8 type V	4-WB	4,300	488 ft
24.					55.	71 of 8 type V	5-WB	8,000	610 ft
25.					56.	71 of 8 type V	3-WB	5,700	315 ft
26.					57.	71 of 8 type V	3-WB	5,100	366 ft
27.					58.				
28.					59.		71WB	25,200	2,074 ft
29.					60.				
30.					61.				
31.					62.				

TOTALS

Date: 7-8-66

Carrier: DOW

Trailer No: 802203

Shipped 17 (Boxes)
25,200

RF Seal 3790
DOW SN 665713

2.0 mtr/h

Figure 3. Handwritten Rocky Flats Plant waste disposal load list.

[illegible]

Figure 4. Page 1 of computerized Form ID-135, "Radioactive Waste Form."

LOAD LIST NUMBER 71 SINGLE LAYER DATE 3/23/72

THIS LOAD IS CLASSIFIED AS LSA

AND HAS BEEN PREPARED FOR VT

AND CONSISTS OF 84 SD ,AND 0 OTHERS

CONTAINER ID	CONTENTS	GROSS	MREM/HR	ELEMENT	WT.	STORAGE
PREFX SERIAL CODE	WEIGHT	SUR 3 FT	1ST	2ND	LOCATION	
224 210 950	315	-5.5 0.0	0.0	0.0	662	
224 211 900	225	-5.5 0.0	0.0	0.0	662	
224 212 950	265	-5.5 0.0	0.0	0.0	662	
224 213 950	310	-5.5 0.0	0.0	0.0	662	
224 216 950	205	-5.5 0.0	0.0	0.0	662	
224 217 950	280	2.8 -5.5	0.0	0.0	662	
224 218 950	380	1.0 -5.5	0.0	0.0	662	
224 219 950	490	0.0 0.0	0.0	0.0	662	
224 220 950	550	0.0 0.0	0.0	0.0	662	
224 221 950	535	0.0 0.0	0.0	0.0	662	
224 222 950	510	0.0 0.0	0.0	0.0	662	
224 223 950	275	-5.5 0.0	0.0	0.0	662	
224 224 950	385	-5.5 0.0	0.0	0.0	662	
228 1 900	170	-5.5 0.0	0.0	0.0	662	
228 101 900	145	-5.5 0.0	0.0	0.0	662	
228 102 900	155	-5.5 0.0	0.0	0.0	662	
228 106 900	165	-5.5 0.0	0.0	0.0	662	
362 102 900	175	-5.5 0.0	0.0	0.0	662	
1444 102 900	175	-5.5 0.0	0.0	0.0	662	
1444 105 900	135	-5.5 0.0	0.0	0.0	662	
1483 105 900	160	-5.5 0.0	0.0	0.0	662	
1483 113 900	155	-5.5 0.0	0.0	0.0	662	
1483 115 900	150	-5.5 0.0	0.0	0.0	662	
1483 116 950	615	-5.5 0.0	0.0	0.0	662	
1483 117 950	665	-5.5 0.0	0.0	0.0	662	
1483 118 950	545	-5.5 0.0	0.0	0.0	662	
1483 122 950	610	-5.5 0.0	0.0	0.0	662	
1483 127 950	360	-5.5 0.0	0.0	0.0	662	
1483 128 950	365	-5.5 0.0	0.0	0.0	662	
1544 311 900	139	-5.5 0.0	0.0	0.0	662	
1544 317 900	152	-5.5 0.0	0.0	0.0	662	
1544 326 900	136	-5.5 0.0	0.0	0.0	662	
1544 329 900	170	-5.5 0.0	0.0	0.0	662	
1544 330 900	145	-5.5 0.0	0.0	0.0	662	
1544 331 900	155	-5.5 0.0	0.0	0.0	662	
1544 332 900	175	-5.5 0.0	0.0	0.0	662	
1544 334 900	150	-5.5 0.0	0.0	0.0	662	
1544 335 900	140	-5.5 0.0	0.0	0.0	662	
1544 337 900	150	-5.5 0.0	0.0	0.0	662	
1544 345 950	640	4.0 0.6	173.5	0.3	R062	

92-5315
 Shipped
 3-27-72

84.5891
 27,104⁹ 4.7 Kg-0-235
 2,334.81 Kg-0-238

SN 72-65B
 HP 357

Figure 5. Page 2 of the computerized load list.

8. NUCLEAR CRITICALITY SAFETY

At RFP, nuclear criticality safety limits for plant operations, storage, and onsite and offsite transportation for fissile materials were established and audited by the Nuclear Criticality Group according to (1) manuals from the AEC, Energy Research and Development Administration, and DOE and (2) transportation and packing regulations from the Bureau of Explosives, Interstate Commerce Commission, and DOT. Plant operational criticality limits were set differently for wet versus dry material. The wet limits were significantly lower than the dry limits. However, adverse experiences in the 1954-1956 timeframe with liquid-leaking drums during transport to INL precluded shipment of wet waste materials.

8.1 Storage Criticality Limits

The main purpose of providing the nuclear criticality limits for plant storage is to differentiate these limits from nuclear criticality limits for transportation, which were more restrictive for fissile material content per container. A secondary purpose is to establish that plant storage criticality limits were set by the plant criticality group, while transportation criticality limits were established by government agencies.

During the 1950s and early 1960s—before construction of the waste storage and loading facility (Building 664)—waste to be shipped offsite was stored outside. During the late 1960s, indoor storage was established to preclude outdoor contamination; however, because of the lack of indoor storage facilities at that time, recoverable SNM in drums also was stored outside. Consequently, material stored outside may or may not have been shipped offsite.

The earliest documented (November 19, 1962) criticality limit discovered is in a letter, “Outside Storage for Drums of Building 71 Washables” (see Appendix J). Typical washables were rags, paper, plastics, and rubber. Washables were processed by water leaching to remove surface contamination. The water-leached washables were then dried and packaged for offsite shipment.

On March 28, 1962, a letter was issued stating the criticality limits for “Storage of 55-Gallon Drums in Buildings 71, 76, and 34” by the manager of the Nuclear Criticality Group (see Appendix K). The majority of these stored drums was processed to recover the plutonium.

Criticality limits for storage of CWS filters in Buildings 70, 80, and outside were issued in a letter by the Nuclear Criticality Group on August 12, 1965 (see Appendix L). The shipment of CWS filters with high-plutonium content in 1965 was most unlikely as plutonium recovery processes were in place.

8.2 Transportation Criticality Limits

Building 664 served as a loading facility for ATMX-600 railcars and for truck trailers. The nuclear criticality limits for storing waste drums and crates reflect the criticality limits for shipping offsite (see Appendix M.).

Offsite shipping containers are more definitively described with issuance of DOT “Special Permit No. 5948” on December 23, 1968, and as amended on January 30, 1969; February 5, 1969; and March 13, 1969.

In the 1950s and early 1960s, a crate limit for fissile material of 15 g/ft³ was generally accepted. The exact date when this criticality limit was reduced to 5 g/ft³ is unavailable. The earliest documentation for 5 g/ft³ is defined in DOT Special Permit 5948, dated March 13, 1969 (see Appendix N).

A letter of August 21, 1967, from E. H. Lee of the Dow Chemical Company (see Appendix O) requests approval for shipping waste in 55-gal steel drums and addresses average plutonium values for drums shipped in 1966 and 1967. See Table 15 for plutonium averages for the drums shipped.

Table 15. Average plutonium values for drums shipped.

Category A	Non-line-generated waste for 1,582 drums with an average plutonium content of 0.024 g per drum
Category B	Line-generated waste for 944 drums with an average plutonium content of 30.8 g per drum
Category C	Building 774 output, Series 741 first-stage sludge for 398 drums with an average of 5.6 g per drum
742	Second-stage sludge, average plutonium content <1 g per drum
743	Grease sludge, average plutonium content 1 g per drum
744	Neutralized hydrochloric acid and average plutonium content of other liquids <1 g per drum
745	Dried salts, average plutonium content <1 g per drum

9. PROCESS AND PLANT CHANGES

Characterization of the waste shipped to INL has changed because of mission, process, and plant changes and additions. The waste shipped to INL can be characterized generally based on plant operations and activities. The majority of the waste generated by plant operations and activities falls into five categories: (1) housekeeping waste, (2) maintenance waste, (3) process waste, (4) major contamination incident waste, and (5) mission changes. This chapter describes this waste and changes from the 1950s through the 1980s.

9.1 Housekeeping Waste

Housekeeping waste results from operating a facility that houses and handles radioactive materials. Examples of housekeeping waste are surgical gloves, cotton gloves, glove-box gloves, paper, plastic bags, rags, contaminated clothing, wood, tape, and other combustible materials. Over the years, housekeeping waste has remained fairly constant, with variations in quantity associated with production and R&D levels. The rising costs of waste disposal stimulated a waste reduction effort for housekeeping waste. The only notable change was an increase of polyethylene-based plastics and a reduction of PVC plastics.

9.2 Maintenance Waste

Maintenance waste was generated by repair and replace activities such as removal of obsolete equipment, installation of new equipment, upgrading safety systems, area equipment removals, and preventive maintenance requirements. These maintenance activities remained fairly constant over the active life of the plant.

9.3 Process Waste

Process waste was generated by five general operations: (1) foundry and fabrication, (2) component assembly and return disassembly, (3) production support activities such as R&D analytical and metallurgical laboratories and inspection, (4) chemical recovery and metal recycle, and (5) waste treatment.

9.3.1 Foundry and Fabrication Waste

In the 1950s and early 1960s, plutonium components were cast and then machined to final configuration. Component casting was eliminated and replaced with rolling and forming to produce components for final machining. Eliminating component casting reduced graphite mold waste but required plutonium ingot casting for rolling stock. Consequently, graphite mold waste remained constant and varied quantity-wise based on mission requirements. In the 1980s, reusable ingot molds were introduced to reduce graphite mold waste.

9.3.2 Component Assembly and Return Disassembly

The waste generated by component assembly and return disassembly activities was minimal when compared to other operations. The disassembly of returns generated classified waste, which was shipped offsite to Hanford and Nevada Test Site. The introduction of new assembly techniques had little effect on waste generation.

9.3.3 Production Support Activities

Laboratory and inspection support operations generated a small quantity of waste that was minimal compared to the total waste produced. These support operations remained fairly constant throughout the production life of the plant.

The R&D effort grew as production requirements became more complex. Waste generated by R&D activities varied according to mission requirements but did not contribute significantly to total waste produced and shipped. The R&D and special-order work generated small amounts of waste with radionuclides such as Np-237, U-233, and others; however, these activities were fairly constant from 1960 to 1989. Consequently, no significant changes occurred.

9.3.4 Chemical Recovery and Metal Recycle

Chemical recovery and metal recycling processes generated significant quantities of waste; however, these processes did not change greatly during the production life of the plant. Chemical recovery processes were nitric-acid based using stainless steel and glass equipment. The plutonium purification process used a tributyl phosphate-kerosene extraction process in the 1950s. This purification process was replaced by an ion exchange process in the early 1960s. The changeover resulted in shipping spent ion exchange resin to INL but eliminated tributyl phosphate and kerosene waste.

Introducing pyrochemical processes in the 1960s generated mixtures of chloride and fluoride salts. The MSE process for the removal of Am-241 from returned plutonium components generated large amounts of waste. Additional pyrochemical development and pilot plant operations generated additional spent pyrochemical salts in the 1970s and 1980s.

9.3.5 Waste Treatment Waste

Waste treatment operations generated two types of waste: liquid and solid. The Liquid Waste Treatment Plant, Building 774, produced sludge from aqueous solutions through a process of ferric-hydroxide-carrier precipitation. This process was used during the life of Building 774 with very little process change. This process was used also in the new Liquid Waste Treatment Plant, Building 374. Consequently, Series 741 and 742 types of sludge were practically the same over time, the only difference being less water content in the Building 374 sludge.

The treatment of organic solutions began in the mid-1960s using a calcium silicate process to produce an organic-based (Series 743) sludge, a waste product of the Building 774 Grease Plant. The calcium silicate process was replaced by an improved solidification process called OASIS. The OASIS process employed a polymeric substance called Envirostone with an appropriate emulsifier. This process was installed in the mid-1980s.

The process solvents changed with requirements of RCRA. Consequently, solvents listed in RCRA were phased out in the 1970s in favor of unlisted solvents.

Solid waste treatment remained reasonably constant in its constituents. The emphasis was on volume reduction and improved packaging. One of the improvements established in the 1970s was the water washing of leaded glove-box gloves to remove any lead-nitrate complexes. The flammability of leaded dry-box gloves in a nitric acid environment has been reported by Johnson and Lindsay (1969).

The most significant advancement in solid waste treatment was waste segregation. A simple five-type designation described the waste shipped to INL during the 1950s and 1960s. In the 1970s, the waste was described using item description codes. The NDA requirements demanded a more homogeneous matrix, which resulted in 88 description codes for waste; however, the aggregate waste shipped did not exhibit major changes, except as noted above.

9.4 Major Contamination Incidents

Rocky Flats Plant experienced two major contamination incidents resulting from two fires. The first fire occurred in 1957 in the R&D areas of Building 771. Cleanup and equipment removal generated mainly Type I combustibles, Type III CWS filters, and Type V noncombustibles. A second fire occurred in 1969 in the Building 776 plutonium foundry. This fire was much more extensive than the 1957 fire; cleanup and equipment removal generated large quantities and various types of waste.

9.5 Mission Changes

The original mission of RFP included manufacturing components of DU, HEU (Oralloy), and plutonium, coupled with pit assembly.

In 1962, the Oralloy mission was terminated, and the plutonium mission was expanded. Cleaning out the Oralloy plant (Building 881) was completed in early 1965. Consequently, significant quantities of Oralloy waste were generated from 1962 to 1965. After 1965, Oralloy waste was minimized.

Building 444 was originally assigned the mission of fabricating components of DU. In the late 1950s, fabrication of beryllium components was introduced and slowly replaced depleted production of uranium components. Consequently, straight DU waste was slowly reduced during the 1960s with only minor quantities generated during the 1970s and 1980s.

During the 1960s, uranium alloys such as uranium-niobium and uranium-titanium were introduced into Buildings 444 and 883. These alloys became the major source of DU waste in the 1960s, 1970s, and 1980s. These alloys and their respective uses are addressed in Section 4.4.6.

The demand for Am-241 by the ORNL Isotope Pool initiated the chemical recovery of Am-241 from the returned plutonium components. Rocky Flats Plant recovered Am-241 in small kilogram quantities until the early 1980s. The need for Am-241 then dropped drastically, thereby terminating recovery processing. By directive from DOE, the Am-241 was sent to the Series 741 and 742 sludge waste streams. In the late 1980s, however, the americium and plutonium in the MSE salts were removed by an aluminum alloy process and shipped to Savannah River Site. Consequently, in the early and mid-1980s, a higher level of Am-241 was present in the aqueous-based sludge from Building 774.

10. SPECIAL-ORDER WORK

In addition to plant mission assignments, requested special-order work was funded directly by purchase orders. The majority of special-order work was requested by the three design agencies: (1) Los Alamos National Laboratory, (2) Lawrence Livermore National Laboratory, and (3) the two Sandia Laboratories. Special-order work generated waste that was not typical of mission production waste.

10.1 Radionuclide Tracer Program

A variety of radionuclides was used for nuclear device diagnostics by Lawrence Livermore and Los Alamos National Laboratories. Nuclear devices fabricated at RFP for test shots at Nevada Test Site were often traced with certain radionuclides. The most prevalent radionuclide used was Np-237 followed by U-233. Plutonium-238, Pu-242, Cm-244, and Am-241 were employed occasionally but not to the extent of Np-237 and U-233.

Radionuclide tracers were added to plutonium and Oralloid metal in minor quantities to form a tracer alloy. The tracer alloy was in a feed ingot configuration, which was analyzed for tracer concentration and was then used to form the part ingot. The part ingot was rolled, formed, and machined to a final configuration.

Tracer alloy preparation procedures generated scrap and residues that could not be processed by the routine processes for plutonium recovery and metal recycling. Consequently, the plutonium-tracer scrap and residues were processed by R&D Special Recovery or sent to Savannah River Site.

10.2 Neptunium Program

The neptunium program at RFP began in 1964 and terminated in 1988. Neptunium-237 was introduced into uranium (DU and HEU) ingots in a 2–10 wt% range for shipment to the ORNL Y-12 plant. This alloy process will not be addressed, as this effort was very limited and generated a minimum of radioactive waste.

10.2.1 Neptunium Acquisition

Neptunium-237 in an oxide form was purchased by Lawrence Livermore National Laboratory from the ORNL Isotope Pool. The oxide was shipped to RFP and delivered to the Chemical Technology R&D Group. For the duration of the program, plutonium-neptunium oxide residues were shipped to Savannah River Site for plutonium and neptunium recovery. Consequently, Savannah River Site was a secondary source of neptunium.

10.2.2 Neptunium Inventory

Yearly inventory of Np-237 at RFP varied from 29 to 1,318 g from 1963 through 1988. For each fiscal year-end inventory, see Table 16. Based on the fiscal year inventories in Table 16, a 5–10% loss to waste would not represent a significant quantity of neptunium sent to INL.

Table 16. Neptunium-237 fiscal year-end inventories.

Fiscal Year	Neptunium-237 (g)	Fiscal Year	Neptunium-237 (g)	Fiscal Year	Neptunium-237 (g)
1963	29	1972	788	1980	744
1964	601	1973	768	1981	486
1965	1,292	1974	470	1982	699
1966	740	1975	485	1983	869
1967	1,215	1976	485	1984	1,040
1968	972	1976A	468	1985	931
1969	1,190	1977	458	1986	985
1970	1,105	1978	567	1987	995
1971	1,318	1979	492	1988	970

10.2.3 Neptunium Material Balance Area Accounts

The material balance area accounts that handled and processed neptunium material are listed in Table 17. Not all of the listed material balance accounts generated radioactive waste sent to INL.

Table 17. Neptunium material balance area accounts.

Material Balance Area	Building	Calendar Years	Material Balance Area	Building	Calendar Years
361-31	559	1973–1984, 1986–1988	1371-29	771	1977
383-32		1986–1988	1371-31		1974, 1976, 1977, 1985–1988
1375-10	371	1985, 1986	1371-42		1973–1988
1375-37		1986	1371-43		1985
1375-50		1984–1988	1374-31		1974, 1975–1986
1375-70		1983–1988	1374-34		1974–1978
1375-80		1985–1988	1374-50		1977–1979, 1984, 1985
1177-07	707	1984	2418-34		1974, 1975
1476-07		1973, 1974, 1977, 1978, 1985	1371-41	776	1988
1576-07		1974, 1977, 1978, 1983, 1984, 1985	1373-76		1988
214-78	771	1980, 1981, 1982	1576-76		1966–1972, 1973, 1974, 1976, 1977
215-80		1979, 1980, 1981, 1982, 1983			1982–1985

Table 17. (continued).

Material Balance Area	Building	Calendar Years	Material Balance Area	Building	Calendar Years
217-40		1983	215-76	777	1986–1988
217-53		1974–1977	223-77		1977, 1978
217-57		1974–1977	1177-38		1982, 1983
217-72		1974, 1975	1177-53		1974, 1977, 1978, 1982–1988
217-80		1974–1980, 1985–1988	1177-81		1983–1988
218-71		1963–1975	214-74	779	1985
218-72		1965–1975	214-79		1984–1987
218-75		1974, 1975	215-79		1984, 1986–1988
218-82		1965–1975	223-79		1978
223-71		1973, 1974, 1977–1982	1374-79		1988
223-73		1978	223-79	779A	1973, 1978
367-31		1963–1987	1144-18	991	1984, 1985
383-33		1973, 1976–1988	1144-40		1976, 1978, 1982, 1984–1986, 1988
1371-06		1985–1987	1144-91		1979–1981, 1983–1986, 1988
1371-27		1988			

10.2.4 Plutonium-Neptunium Alloy Preparation

Two methods of preparing a plutonium-neptunium alloy were developed. The first method was a co-reduction method in which plutonium oxide and neptunium oxide were mixed and hydrofluorinated to produce their respective fluoride compounds. The mixed fluorides were reduced with calcium metal to form an alloy button. The button was then cast with plutonium metal feed to produce a feed ingot with the required concentration of neptunium.

The second method prepared neptunium metal by hydrofluorinating neptunium oxide to produce a neptunium fluoride, which was then reduced with calcium to produce a neptunium metal button. The button or part of the button was cast with plutonium feed metal to form a feed ingot.

At times, a short ingot was cast and analyzed for neptunium content. A second feed ingot casting was prepared using the short ingot plus the proper amount of plutonium metal feed to produce the specified neptunium concentration.

The co-reduction procedure was initially employed but was discontinued in favor of the neptunium metal addition method. The co-reduction procedure generated more residues and did not consistently produce the desired concentration of neptunium.

10.2.5 Scrap and Residue Generation

Both alloy preparation methods generated scrap and residues that could not be put through the routine processes for recovering and recycling plutonium. Consequently, the plutonium-neptunium scrap and residues were processed by R&D or Special Recovery or sent to Savannah River Site.

Casting, forming, and machining activities generated plutonium-neptunium metal scrap. When feasible, alloy scrap was cast into ingot form for reuse. If the Am-241 content was >50 ppm, the ingot was subjected to MSE to reduce the americium content to <50 ppm. The alloy scrap was stored and was not sent to INL. Three types of residues were generated: (1) mixed oxide; (2) sand, slag, and crucible; and (3) molten extraction salts.

10.2.5.1 Mixed Oxide Residues. The metal alloy chips, turnings, and casting skull were burned to an oxide form. The mixed oxide was processed by nitric acid dissolution followed by ion exchange to mainly recover the plutonium and salvage as much of the neptunium as feasible. However, a small amount of the neptunium was sent to Building 774 in the form of nitrate and chloride solutions. The recovered plutonium in nitrate solution form was transferred to the War Reserve plutonium stream. The neptunium sent to Building 774 was probably in the 1–10 g range.

10.2.5.2 Sand, Slag, and Crucible Residues. The sand, slag, and crucible residue generated by the co-reduction procedure was leached with nitric acid to recover the plutonium and neptunium. The nitrate solution was transferred to the ion exchange procedure described above. The sand, slag, and crucible residue from the neptunium metal reduction procedure was processed by R&D to recover the neptunium in oxide form. The leached sand, slag, and crucible residue was sent to INL if it met the economic discard limit for plutonium only.

10.2.5.3 Molten Extraction Salt Residues. The major quantity of spent molten extraction salts, consisting of a mixture of magnesium, potassium, and sodium chloride salts, was stored for future recovery. The plutonium content as a chloride averaged about 3%; however, R&D developed a limited recovery procedure. Consequently, only milligram quantities of neptunium were sent to Building 774 in chloride and nitrate solutions. No molten extraction salts involving neptunium were sent to INL.

10.2.5.4 Graphite Molds. Casting graphite molds were processed for plutonium recovery through scarfing and nitric acid leaching of the scarfings. Scarfed graphite molds were sent to INL based on the economic discard limit for plutonium. Likewise, leached graphite scarfings were sent to INL based on the plutonium economic discard limit for graphite fines. Consequently, neptunium content in the graphite was in the microgram range.

10.2.6 Waste Generation

Liquid waste was sent to Building 774 and was a constituent in first- and second-stage sludge. Organic liquids also were sent to Building 774 to be processed by the Grease Plant. Considering the dilution factor in Building 774, neptunium concentrations in Series 741, 742, and 743 sludge were insignificant.

Combustible waste was generated but not segregated for recovery, since a significant segment of combustible waste was not line generated. The neptunium content of non-line-generated waste was probably not detectable. The majority of line-generated combustible waste was incinerated and the ashes leached for plutonium recovery. The resulting nitric acid solution was sent to ion exchange for plutonium recovery. Any neptunium present would have been sent to Building 774 in liquid form.

10.2.7 Summary

The number of events at Nevada Test Site that used Np-237 ranged from one to four per year with two per year being the norm. Consequently, the quantity of neptunium shipped to INL compared to plutonium waste can be considered insignificant. However, alpha decay of Am-241 to Np-237 also should be taken into account.

The majority of the neptunium sent to INL was in Series 741, 742, and 743 sludge. Negligible amounts were sent through graphite and sand, slag, and crucible residue.

10.3 Uranium-233 Program

Rocky Flats Plant was assigned the mission for U-233 production. Consequently, requests for fabricating U-233 items were referred to RFP. However, requests received were not mission related, but on a special-order basis.

The majority of U-233 work was associated with device-testing projects at Nevada Test Site. Uranium-233 often served as a tracing material for device-testing diagnostics. The chief requestor was Lawrence Livermore National Laboratory.

Uranium-233 was received from ORNL in oxide form. The U-232 content varied from several hundred parts per million to less than 100 ppm. The decay scheme for U-232 produces daughter products with high gamma emissions. To interrupt the decay sequences, a thorium strike was performed to remove the Th-228 daughter.

General processing consisted of dissolution of the oxide in nitric acid followed by a thorium strike using fluoride ions. Uranium-233 was precipitated as a peroxide, calcined to uranium oxide, hydrofluorinated to uranium tetrafluoride, and reduced to uranium metal with calcium metal and an iodine booster. Resulting metal was cast and machined to the required shape(s).

10.3.1 Scrap and Residues

Machining scrap was burned to uranium oxide. Metal reduction residues were leached with nitric acid for uranium removal. Uranium was precipitated with ammonium hydroxide and calcined to uranium oxide. The oxide was shipped back to ORNL.

10.3.2 Waste Generation

Items that did not contain significant quantities of U-233 were declared waste and were shipped to INL. All combustible waste was shipped. Other items that may have been shipped were glassware, small process equipment items, filters, and other miscellaneous items. Leached reduction residues and graphite casting molds were shipped to INL.

Liquid waste was transferred to Building 774, the Liquid Waste Treatment Plant. Small amounts of U-233 were blended into the Series 741 sludge. Higher levels of U-233 were processed as cemented liquids if necessary. Lead-lined drums were used if necessary to meet the requirement for contact handling.

10.3.3 Processing Facilities

The U-233 special orders were carried out in the Oralloy plant located in Building 881 in the 1950s and 1960s. Decommissioning of Building 881 moved the U-233 special-order work into the plutonium processing facilities. In the 1970s and 1980s, chemistry processing was carried out in the R&D area in Building 771. Casting and machining took place in R&D metallurgical facilities in Buildings 771 and 776.

Plutonium glove boxes were cleaned and scrubbed before introducing U-233. Oak Ridge National Laboratory placed a limit of <30 ppm of plutonium in any residue returned there; however, slightly higher part per million levels were accepted if the returned material could be blended down. If the oxide was contaminated with unacceptable levels of plutonium, it was stored for future use. Other U-233 residues contaminated with plutonium were shipped to INL.

The U-233 projects were monitored by the Health Physics Group to ensure radiation safety and that the principles of as low as reasonably achievable were followed.

10.3.4 Summary

The U-233 program was not a major effort at RFP. The frequency and scope of the projects were less than one per year with a duration of a month or two; however, discard limits were quite high for U-233 as reprocessing capability and capacity were limited.

10.4 Americium Program

The beta decay of Pu-241 to Am-241 provided a source for a single isotope of americium. The Pu-241 concentration in War Reserve plutonium was in the 0.2–0.3 wt% range. With a half-life of 14 years for the beta decay of Pu-241, returned pits could have an Am-241 concentration from 500 to 1,500 ppm. The americium was an unwanted diluent for plutonium recycled metal and did not meet metal specification for pit material. Consequently, removal of Am-241 was necessary to obtain a specification of <50 ppm at the time of casting a plutonium feed ingot.

The demand for Am-241 remained high from the late 1950s to the early 1980s. After that, the demand was greatly reduced, and Am-241 was declared a waste product by DOE.

The americium recovery process was carried out in Building 771 with liquid waste solutions treated in Building 774.

10.4.1 Americium Feed Sources

The plutonium purification process used the precipitation of plutonium peroxide from a plutonium nitrate solution using hydrogen peroxide as the precipitating agent. The impurities and Am-241 remain in the filtrate and precipitate wash solution. These two solutions were the original feed source for the americium recovery process.

In 1967, an MSE process was developed that extracted 90% of the americium content from plutonium metal. The MSE process was carried out in Building 776. Spent MSE salts were sent to Building 771 for plutonium and americium recovery.

The MSE process reduced the americium content in the plutonium peroxide filtrate. The americium content finally reached a level that was not economically feasible to recover. Consequently, filtrate processing was discontinued. The MSE salts then became the sole source of americium feed.

10.4.2 Americium Recovery Processes

The history of the americium recovery process is well presented by Beach and Perry (see Appendix P) in an internal report. This report correlates process changes with dates and indicates processing efficiencies accordingly. For detailed information, consult the Beach and Perry report in Appendix P.

10.4.3 Waste Streams

The majority of waste streams were liquids transferred to the Liquid Waste Treatment Plant Building 774. The report by Beach and Perry (see Appendix P) describes these waste streams.

Solid waste generated was spent anion and cation ion exchange resins, filter pads, and general housekeeping combustibles. Some glassware and other similar noncombustible processing equipment were discarded also. All discards were based on plutonium content regardless of their respective americium content.

10.5 Thorium Program

The thorium program was very limited in scope and was supported by special-order work. Thorium work—natural thorium does exhibit radioactivity—was carried out by the technical staff, which was considered to be an R&D organization. The thorium program was mainly a casting, rolling, and fabrication effort. Thorium scrap was returned to ORNL. A very limited amount of combustibles was generated and sent to INL.

The thorium work was carried out mainly in Buildings 331 and 881 during the early 1960s. No records were searched to identify the thorium work more precisely because the program was limited and had few progress reports available.

10.6 Curium-244 Program

The Cm-244 program was limited to a duration of about 6 months. Curium-244 was introduced into plutonium using the co-reduction technique. The curium-plutonium short ingot was blended into a feed ingot to obtain the concentration of specified parts per million for the Cm-244. The curium-plutonium scrap and residues were stored pending shipment to the Savannah River Plant. Line-generated combustibles and secondary residues were shipped to INL based on their plutonium content.

10.7 Other Special-Order Work

The special-order work reported above generated waste with unique characteristics and constituents compared to regular mission-generated waste. However, significant special-order work that did generate waste typical of regular mission-generated waste was performed throughout the productive life of RFP; this form of special-order work is not addressed in this report.

11. CLASSIFIED WASTE

Rocky Flats Plant was assigned the mission to disassemble returned pits. This mission generated non-SNM classified pit components contaminated with trace amounts of plutonium and Orallo. In addition, rejected and surplus non-SNM classified components added to the backlog of contaminated disassembled components.

Pit foundry and manufacturing processes produced contaminated classified tooling, fixturing, gauging, casting molds, plastic templates, and plastic shapes. The level of pit disassembly was governed mainly by the demand for plutonium to support new weapon builds and by obsolescence factors.

Pit production urgencies coupled with no significant declassification facilities and limited storage capacity prompted RFP to ship the contaminated classified items to Hanford and Nevada Test Site.

11.1 Shipment of Contaminated Classified Material

Rocky Flats Plant was authorized by the AEC, Energy Research and Development Administration, DOE, and sites listed in Table 18 to ship contaminated classified materials to their respective repositories.

Table 18. Classified waste shipment sites.

Site	Time Period
Idaho National Laboratory	1954–1964
Hanford	1958–1984
Nevada Test Site	1958–1964 1985–1989

In 1954, RFP received authorization from AEC Idaho Operations Office and Phillips Petroleum Company (site AEC contractor) to ship solid radioactive waste to the National Reactor Testing Station burial ground at INL. The authorization included contaminated classified waste.

Classification questions were raised by the AEC Idaho Operations Office and Phillips Petroleum that related to the isotopic composition of weapons-grade plutonium and the enrichment level of DU, which were classified at that time. Correspondence by classification personnel confirmed classification of the plutonium isotopic composition and enrichment level of DU; however, general low-level contaminated plant waste sent to INL continued as unclassified shipments. The reasoning associated with this decision was not addressed in the historical correspondence reviewed. The isotopic composition of weapons-grade plutonium was declassified in April 1964, and the enrichment level of DU was declassified shortly thereafter.

The AEC courier receipts were obtained for 1963 and early 1964 indicating the shipment of classified material to INL (see Appendix Q). Unfortunately, the courier receipts do not identify the contents of the classified material. At that time, RFP had a large backlog of low-level plutonium residues that exceeded the economic discard limit. Rocky Flats Plant requested permission from the AEC Albuquerque Operations Office to send the residues to INL for burial. Permission was granted, but RFP was required to provide health physics escorts for safety reasons.

The courier receipt in Appendix Q indicates that the radioactive waste is classified. The corresponding waste shipment data sheets for Trailers TZ-20 and TZ-21 (see Appendix R) do not indicate that classified shapes were included. The load lists for Trailers TZ-20 and TZ-21 (see Appendix S)

indicate that the types of waste were mainly combustibles (Type I) and sludge (Type IV). However, noncombustibles (scrap metal) are indicated by Type V or Type 5 for two drums originating from Buildings 776 and 771, respectively, for Trailer TZ-20. The load list for Trailer TZ-21 indicates only combustibles (Type I) and sludge (Type IV). There is the possibility that the two drums of Type 5 scrap metal were classified shapes. However, the normal operating practice for loading trailers would be to store the classified drums until a full trailer load was available. Consequently, it is highly unlikely that the two Type 5 drums contained classified shapes.

The higher quantities of plutonium may have been the reason for using the AEC courier system. Classified courier shipments were terminated in 1965. The declassification of plutonium in April 1964 also may have contributed to terminating these classified courier shipments.

The INL personnel have repeatedly inquired about the possibility that classified shapes have been sent to INL. A search of trailer load lists, ATMX-600 railcar load lists, waste shipping memos, 741 forms, and traffic correspondence provided no direct evidence that classified shapes were shipped to INL. Inquiries of former and present RFP waste and traffic personnel revealed no recollection of classified shapes being sent to INL.

A letter from the Dow Traffic Group to the Nuclear Engineering Company states that no classified waste was shipped to INL during the period from July 1963 through May 1964 and confirms that the classified waste was sent to Hanford during this period (see Appendix T). However, there is the possibility that a stray classified item may have been shipped inadvertently in an unclassified container.

12. SPECIAL TOPICS

Communications with various INL personnel revealed specific topics of interest that are addressed in this section.

12.1 Graphite Waste

Graphite waste was the result of foundry casting operations. Graphite molds were used for casting DU, HEU, and plutonium into various metal forms. The molds were fabricated in a carbon shop located in Building 445, which was attached to Building 444.

Before casting, a coating was applied to the mold surface to prevent reaction of the graphite with the molten metals. The most prevalent mold coating was calcium fluoride followed by yttrium oxide. Other oxide-based mold coatings were used experimentally.

Spent molds were often scarfed to remove any residual metal or metal that may have breached the mold coating. The HEU and plutonium scarfings were leached with nitric acid to recover the uranium and plutonium. The leached scarfings that were really graphite fines were packaged into a suitable container and placed in a drum for shipment to INL. Not all spent molds were scarfed if they were below the economic discard limit.

Scarfings from the DU molds were calcined to convert the uranium metal to an oxide form. Calcined material was packaged and placed in a drum for transport to INL. Rocky Flats Plant did not have chemical recovery facilities for recycling DU. Converted uranium oxide was often identified as “RO” on the trailer and ATMX-600 load lists.

Graphite shipped to INL had a variety of physical forms (e.g., there were whole molds, fines, pieces, chunks, and partial molds). Undoubtedly, a small amount of fines was present with any of the solid graphite mold configurations. The graphite stock employed to fabricate the molds was a high-density, nuclear-grade extruded graphite (see Appendix U).

12.1.1 Plutonium Graphite Molds

The Zodtner and Rodgers (1964) report has caused concern at INL because of the significant quantity of plutonium arbitrarily assigned to graphite molds in the 1950s and early 1960s. Since INL is concerned primarily with plutonium molds, no further discussion of DU and HEU molds will be included in this section.

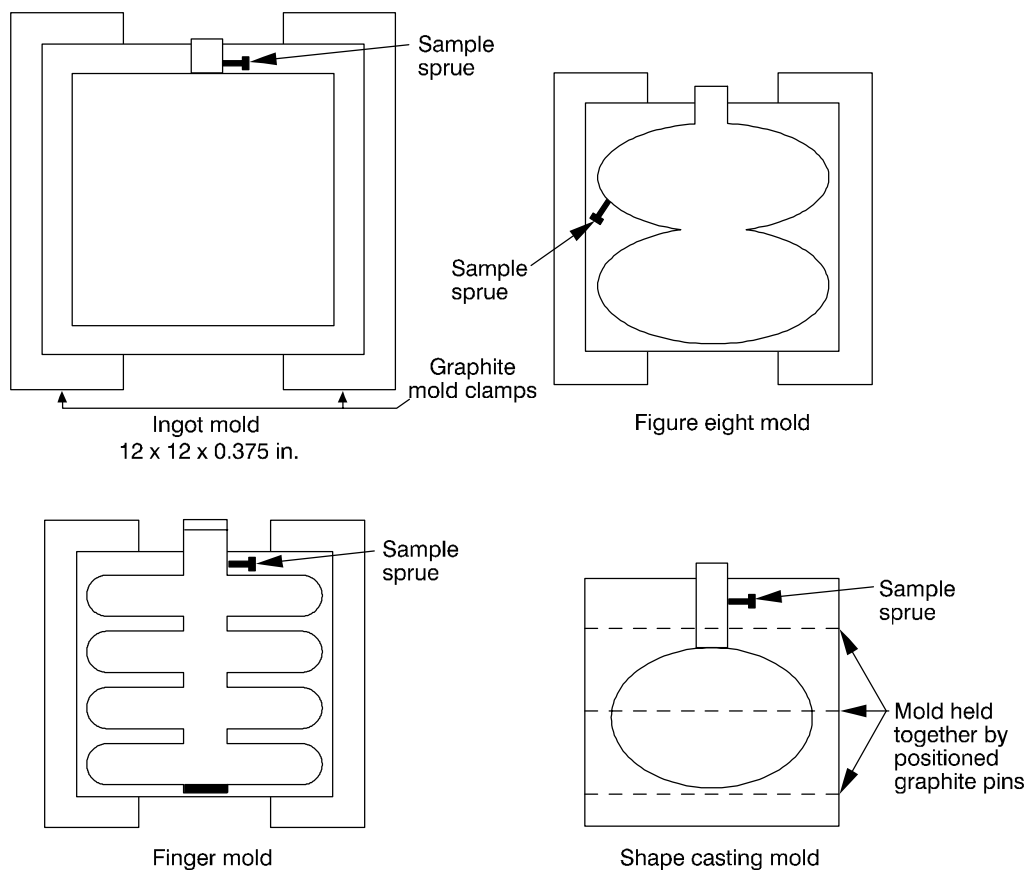
12.1.1.1 Generation Rates. The early shipments of radioactive waste to INL were defined by five designated types. Graphite molds and segments were assigned a noncombustible Type V. The noncombustible Type V included scrap metal, glass, ceramics, and graphite. A drum listed as Type V could contain a mixture of noncombustible material. Consequently, this system precludes identifying graphite mold waste through shipping records. Available waste documentation does not include shipping records for graphite molds, nor do records of building operations indicate generation rates of graphite mold waste.

This nomenclature continued until the late 1960s, when a new system of material designation was introduced using a digital system of item description codes. Introducing this system was driven by the development of economic discard limits and nondestructive radiometric instrumentation (i.e., drum and can counters). The system of item description codes precluded commingling noncombustible waste and provided a way to record shipments of graphite waste to INL.

From 1953 to 1956, plutonium foundry operations were carried out in Building 771 on a limited scale. Plutonium casting operations used a split copper mold configuration. Consequently, very little (if any) graphite waste was generated.

In 1957, Building 776 came into operation and began using graphite molds. The initial graphite molds were porous and of low density. High-density, nuclear-grade graphite later replaced the less dense graphite in foundry operations.

12.1.1.2 Types of Plutonium Graphite Molds. For War Reserve production, the four general configurations of molds are illustrated in Figure 6.



G1412-01

Figure 6. Four general configurations for graphite molds.

The above mold dimensions and the location and number of sample sprues would change somewhat for different weapon programs, although the general mold shapes would remain fairly constant.

Most of the plutonium molds were associated with War Reserve mission work; however, special-order work and R&D mold experimentation generated a minor quantity of molds with a variety of configurations. Since the molds had to conform dimensionally to the casting furnaces, the outer dimensions were reasonably constant.

12.1.1.3 Foundry Casting Operations. The graphite molds were coated with calcium fluoride to control plutonium penetration into the graphite mold. Later, yttrium oxide was used also as a mold

coating. The split molds were clamped using graphite clamps, which were discarded when defective. Shape molds varied in configuration and were clamped using graphite pins.

These molds were constructed to fit into vacuum-induction casting furnaces and to accept a molten charge of plutonium metal. On cooling, the mold was removed from the furnace and transferred to a break-out glove box. The mold was disassembled and the plutonium item removed. The sample sprue was cut off and packaged for transfer to the analytical laboratory for elemental analysis and plutonium assay. Part of the sample sprue was saved, should reanalysis be required. The surface of the plutonium item was brushed to remove any loose particles. The plutonium item was then transferred to the next scheduled operation. Any identifiable plutonium pellets and casting slag were collected and placed in a container for transfer to plutonium recovery in Building 771.

The brushings, glove-box floor sweepings, and collected graphite fines were added to the mold drum. The plutonium content added to the drum was estimated by using by-difference techniques. When the drum reached approximately 500 lb, the drum was sealed and sent to waste disposal.

In 1965, an experimental drum counter became available for measuring the plutonium content of graphite mold drums. The drum counter provided improved assay values and instituted reprocessing for plutonium recovery. These activities necessitated improvements in plutonium waste segregation. As a consequence, mold break-out fines and sweepings were placed in a gallon-sized paint can for plutonium recovery processing. Later, the development of a can counter reduced the collection container to a quart-sized, specially manufactured, stainless-steel can to improve the plutonium assay.

12.1.1.4 Plutonium Estimates on Graphite Molds. The drum counting reports describing graphite assaying indicate that graphite mold waste was given priority over other waste forms. However, other than Zodtner and Rogers (1964), no formal documentation has been located that defines plutonium estimates in graphite molds. An informal reference for the average plutonium content for line-generated waste was 30 g (see Appendix C). Graphite molds are a component of line-generated waste.

The first workable drum counter was completed in 1964 and installed in Building 771. Internal reports describing the counting efforts and results for graphite indicate that the plutonium results in graphite drums were low and required adjustment by a factor of 1.8 ± 0.18 (see Appendixes V and W).

12.1.1.5 Plutonium Recovery for Graphite Molds. As stated previously, spent graphite molds were broken up with a ball-peen hammer and placed in a 55-gal drum with an 8-mil plastic liner. When full (~226.8 kg [500 lb]), the drum was closed and assigned a by-difference plutonium value.

Starting in the mid-1960s, the mold surfaces were scarfed with a needle gun. Surface grinding was also employed. The scarfings and grinding powder were collected for plutonium recovery. The scarfed or ground graphite pieces and chunks were placed in a 55-gal drum for shipment to INL.

The leaching procedure used to recover plutonium from graphite was based on the experimental work reported by Williams and Pinamont (1965). This report also observed that plutonium assays of graphite feeds were low by a factor close to two. The collected scarfing or grinding powders were placed into dissolver pots. The original dissolver pots were glass but later were replaced with Teflon pots with a capacity of 3–4 L (3.2–4.2 qt). Appropriate amounts of nitric acid were added to the pots to dissolve the contents. The nitric acid solution was heated using an electric immersion heater. The dissolved plutonium solution was removed leaving a graphite heel in the bottom of the dissolver pot. The heel was scraped out of the pot and placed in a stainless steel pan. The heel was dried on a hot plate and was removed from the pan using a hammer to break up the solidified graphite material. The removed graphite material was

placed in a plastic-lined 55-gal drum. When the drum was full, the drum was sealed and labeled, and an estimated gram value, usually zero, was assigned to the drum.

In the late 1960s, the leached graphite was packaged into paint cans and was assayed with a can counter. Later, the volume of the leached graphite container was reduced to 0.9 L (1 qt) for an improved plutonium assay. Polyethylene bottles also were used as graphite containers.

The decision to process discarded molds was based on reducing the material unaccounted for. Consequently, scarfing, and grinding procedures were coupled with leaching the graphite removals using nitric acid to recover the plutonium. The scarfed graphite pieces contained minimal quantities of plutonium.

Breaching of the mold coating by the molten plutonium was the chief cause of plutonium loss to the mold. Using low-density graphite molds contributed to the plutonium loss. Using high-density, nuclear-grade graphite for molds helped alleviate the breaching problem.

12.1.1.6 Waste Management of Graphite Molds. Using graphite molds for plutonium casting was implemented in 1957 in Building 776. Before 1957, copper molds were used in Building 771. Plutonium mission requirements were fairly constant from 1957 to 1962 with a modest use of graphite molds. From 1963 to 1970, mission requirements of plutonium components increased significantly, which resulted in a large inventory of spent graphite molds.

The packaging of spent graphite molds from 1957 to 1962 was completed by using either a 5- or 8-mil polyethylene liner inside a 55-gal drum. The type of drum was not standardized, and vendor drums often were used. The increased demand for 55-gal drums resulted in purchasing 17-H and 17-C drums.

The early graphite mold drums were usually considered full when their weight was about 500 lb. Later, a limit of 90.7 kg (200 lb) of graphite per drum became a requirement. The letter from William F. Romine, Traffic Manager, in 1968 quotes a limit of 90.7 kg (200 lb) of graphite per drum for nuclear criticality safety reasons (see Appendix X). A certificate of approval issued in 1969 for fissile-large quantity shipping containers for ATMX-600 railcars issued by the AEC Albuquerque Operations Office quotes a limit of 45.4 kg (100 lb) of graphite for 30-gal drums and 90.7 kg (200 lb) for 55-gal drums for nuclear criticality safety reasons (see Appendix Y).

Graphite molds in the late 1960s and graphite mold segments were scarfed or ground to remove surface plutonium. The surface removals were placed in metal containers and bagged from the glove-box line into 55-gal drums. The scarfed or ground segments were placed directly into the 55-gal drums. This general drum-packaging configuration remained reasonably constant until the concept of retrievable storage—driven by the AEC directive (AEC 1970)—became the rule.

12.1.1.7 Miscellaneous Graphite Waste. Analytical laboratories in Buildings 441, 771, and 881 generated graphite electrodes contaminated with DU, plutonium, and HEU, respectively. The graphite electrodes were used in emission spectroscopy to determine elemental impurities in DU, plutonium, and HEU matrix products. The spent electrode charge was removed from the graphite electrode cup by tapping into a stainless steel vessel. The spent anode graphite cup electrode and the pointed spent cathode graphite rod electrode were placed into a second stainless steel vessel. However, the spent electrode charge was not removed from the graphite anode cup electrode for DU analysis. The collected spent electrode charges for plutonium and HEU were sent to chemical recovery to reclaim the plutonium and HEU. The collected spent electrodes were packaged into the noncombustible (Type V) waste category.

The anode cup graphite electrode was fabricated from a graphite electrode (~0.97-cm [0.38-in.] diameter), while the upper cathode graphite electrode was a pointed graphite rod (~0.33-cm [0.13-in.] diameter). Both electrodes were about 3.81 cm (1.5 in.) long.

To surpass the complicated emission spectra of uranium and plutonium, a carrier was added. The carriers employed were silver chloride, gallium oxide, and sodium fluoride. Consequently, the spent electrodes may be contaminated with these compounds. These spent graphite electrodes were not a significant contributor to the graphite waste shipped to INL.

Other graphite-based items were spent graphite bearings from chemical pumps, graphite pegs to hold molds together, and experimental casting configurations generated by R&D efforts. These items were not significant generators of graphite waste.

12.1.2 Plutonium Discard Limits

In the late 1950s and the early 1960s, RFP recognized that significant quantities of plutonium were being lost through the solid waste streams. This fact dictated the need for improved plutonium measuring systems and the installation of appropriate recovery systems to return the plutonium to the War Reserve production stream and to reduce the plutonium levels in the waste shipments.

In 1960, a project provided plutonium recovery capability and capacity for treating low-level solid waste, including graphite. The project included the development of a waste drum counter. Construction was completed in 1962, and the new recovery systems were placed online in the mid-1960s.

12.2 Roaster Oxide

Depleted uranium operations produced pyrophoric uranium fines, turnings, chips, chunks, and casting skull. Disposal of these pyrophoric items was a constant problem, as no DU recovery facilities were available at RFP. Shipment to other DU sites was not feasible because of their pyrophoric condition. In 1956, a calcining system was constructed to oxidize these pyrophoric materials to a uranium oxide form. This system was upgraded several times during its lifetime into the 1980s.

12.2.1 Roaster Oxide Description

Uranium oxide is a black, fine powder; however, added impurities, such as heat-treating salts, will alter the color from black to a black-gray depending on the uranium oxide content.

Roaster oxide disposal was often abused by including hunks of uranium metal, tooling, work gloves, and other items contaminated with DU.

12.2.2 Chip Roaster

The chip roaster feed was mainly DU and DU alloy chips, turnings, saw filings, and any other source of uranium metal collections. These metallic forms were introduced into a calcining system designated as the chip roaster. Consequently, the resulting uranium oxide powder was called roaster oxide. On shipping lists, it was listed as RO to signify DU oxide. To remove any RCRA constituents, the chips and turnings were steam-cleaned before being introduced into the chip roaster in the 1980s. The operation of the chip roaster was shut down from 1959 to 1961 because of its relocation in Building 444 (see Appendix Z).

12.2.3 Casting Skull

Uranium and uranium-alloy casting skull were burned in a foundry burn box to an oxide form. A large crucible was employed to burn the collected skull material. The burned (oxidized) skull oxide was loaded into a 30-gal drum and labeled roaster oxide.

12.2.4 House Vacuum System

The house vacuum system collected any spilled or extraneous uranium oxide or any other dry powder form. A cyclone separator was positioned in the house vacuum system to remove the powder material from the house vacuum stream. The collected material from the cyclone separator was packaged in 30-gal drums and labeled roaster oxide.

12.2.5 Sintered Metal Filter Collections

Sintered metal filters were located in the chip roaster exhaust system and in other exhaust systems including the house exhaust system. The collected particles on the metal filters were removed and composited and then placed in a 30-gal drum and labeled roaster oxide.

12.2.6 Water-Quenched Tanks

Sludge from the water-quenching tanks was created from heat treating and rolling mill operations in Buildings 444 and 883. The sludge accumulated from surface uranium oxide and residual heat-treating salts. The quenching water was drained and sent to Building 774. The wet sludge was collected, air dried, and packaged in 30-gal drums and labeled roaster oxide or sludge. At times, the sludge collected was introduced into the chip roaster. If the sludge burned, it was labeled roaster oxide. If not, it was labeled sludge.

12.2.7 Furnace Box Stubs

Uranium metal strips were bolted together and heated in an arc melting furnace. The center melt was poured into a mold configuration, while the end portions were discarded as box stubs. The box stubs were combined with the oxide from the chip roaster. The drums with box stubs were identified as roaster oxide.

12.2.8 Packaging Roaster Oxide

The roaster oxide was placed in a 30-gal drum that was overpacked with a 55-gal drum. Vermiculite was added to fill the void space between the two drums. The outer drum was wiped to remove any residual contamination. The 55-gal drum was labeled and prepared for shipment. Later, a plastic 55-gal drum liner and a cardboard disc cover were used in the packaging configuration.

12.2.9 Roaster Oxide Pyrophoricity

Stakebake and Osborn (1994) evaluated the potential pyrophoricity of the roaster oxide, concluding that there was a low probability that the few uranium chips that may be present in a drum of roaster oxide would ignite and if so, the major matrix of oxide would absorb the heat produced to preclude drum rupture.

12.2.10 Roaster Oxide Shipments

A summary of the DU waste shipped to INL from 1954 through 1970 is shown in Table 19. Table content was generated from monthly and annual history reports by the Waste Disposal Coordination Group.

Table 19. Summary of depleted uranium waste shipments to Idaho National Laboratory based on reports by the Waste Disposal Coordination Group.

Calendar Year	55-Gallon Drums	40-Gallon Drums	30-Gallon Drums	Boxes ^a	CWS Filters ^b	Tanks	Total Volume (ft ³)	Gross Weight (lb)	U-238 ^c (kg)
1954	1,217 ^d	—	—	—	—	—	—	—	738
1955	1,564	—	115	—	—	—	12,248	390,104	979
1956	1,795	—	—	—	—	2	12,347	315,727	1,174
1957	1,882	—	300	—	460	—	22,176	863,800	2,147
1958	818	37	220	—	327	—	8,055	283,938	4,209
1959	692	—	97	4	—	—	5,323	200,380	3,753
1960	839	—	28	17	—	—	6,866	230,913	4,123
1961	1,030	—	37	29	333	—	10,236	268,708	4,311
1962	839	—	4	24	—	—	6,775	208,882	4,674
1963	1,510	—	3	24	92	—	12,629	286,966	1,672
1964	2,058	—	—	42	93	—	19,381	386,931	1,339
1965	1,479	—	—	41	—	—	15,742	326,797	4,269
1966	1,488	—	—	31	—	—	14,509	420,113	53,452
1967	1,473	—	—	64	—	—	18,434	498,914	53,176
1968	1,491	—	—	44	—	—	16,216	390,470	33,373
1969	1,087	—	—	40	—	—	13,028	326,098	22,721
1970	567	—	—	63	—	—	11,252	172,383	7,084
Totals	21,829	37	804	423	1,305	2	205,217	5,571,124	203,194

a. The standard size waste box was 4 × 4 × 7 ft. Some boxes of slightly different sizes were shipped.

b. "CWS filter" was terminology used for what are now high-efficiency particulate air filters. Most were 2 × 2 × 1 ft in size, shipped in boxes in early years. In later years, some were shipped in drums.

c. Data on total weight of U-238 shipped came from a separate summary report and were not related to individual containers.

d. The report for 1954 did not break out the drum size, volume, or weight.

CWS = Chemical Warfare Service

12.3 Ion Exchange Resins

Ion exchange resins were used extensively from the late 1950s through 1989. The three major ion exchange resins employed for plutonium recovery were: (1) Dowex 1 × 4 [50-100] mesh, (2) Dowex II [20-50] mesh, and (3) Amberlite IRA-938 [20-50] mesh. These resins were used in a nitrate form. Dowex 50 × 8 cation resin was used in the americium separation process. The major resin used was Dowex 1 × 4 anion resin (nitrate form) with Dowex 50 × 8 cation resin in second place. Amberlite IDA-938 was used in special recovery processing during the 1970s and 1980s. Other ion exchange resins were employed by R&D and the analytical laboratories for evaluation and specific applications. Very limited amounts were involved in these activities.

Spent ion exchange resins were water-washed to remove residual acids. Two disposal methods were used: (1) incineration and (2) mixing with cement for shipment to INL. During the 1960s, spent ion exchange resins were mixed with cement in a 1-1 ratio. Later, the ratio was altered to 1.5 parts resin to 1 part cement as indicated in a letter from William F. Romine (see Appendix AA).

12.4 Decommissioning Building 881

As discussed previously, Building 881 discontinued producing Oralloy (HEU) components in the early 1960s. Decommissioning activities generated significant quantities of Oralloy waste, including capital equipment. Appendix BB illustrates the types of waste generated. The items marked with a date stamp were shipped to INL. Those items not marked were processed internally, stored, or both. The organic liquid waste was stored on the 903 Pad and processed later by the Grease Plant, Building 774.

12.5 Discard Mud

Impure Oralloy-bearing materials were crushed in a rod mill into pea-sized feed. The crushed feed was leached with nitric acid followed by a filtration step. The insoluble residue was called discard mud and had very little U-235 remaining. The major source of the discard mud was sand, slag, and crucible material generated from the bomb reduction of uranium tetrafluoride with calcium metal to form a uranium metal button. The major chemical constituents of the sand, slag, and crucible residue were magnesium oxide sand, magnesium oxide crucible, and calcium fluoride slag. Other materials such as furnace liners (alumina) and uranium chip-burning graphite pots were also introduced to the rod mill operation but in minor quantities. The discard mud was composed of rod mill constituents not soluble in hot nitric acid.

12.6 Plutonium-Contaminated Oralloy Waste

Several items in Appendix CC are listed as contaminated with traces of plutonium. The disassembly of returned pits generated Oralloy components contaminated with surface plutonium. Building 881 removed the surface plutonium contamination with a dilute nitric acid rinse, followed by a water rinse. The leached HEU component was dried and subjected to radiometric scan to determine any plutonium content. Plutonium-free HEU components were returned to the HEU foundry. Later, after decommissioning the HEU foundry, the HEU components were shipped to the ORNL Y-12 plant.

The leach solution was processed in a glass evaporator. The high-acid condensate was stored in a ring-packed tank and recycled to the leaching container as clean acid. The evaporator concentrate was treated with ammonia gas to precipitate the actinides. The precipitate was calcined to an oxide and shipped to the ORNL Y-12 plant if the plutonium content was very low or to Savannah River Site if the plutonium content was too high (Giebel 1964). The leaching and rinsing process generated HEU and

plutonium-contaminated solid waste such as rags, absorbent wipes, filters, and other solid waste, which were packaged and shipped to INL.

Leaching of returned Oralloy was transferred to Building 771 in the 1970s. The HEU leach solution with plutonium contamination was processed in the special recovery area using a modified plutonium and uranium recovery by extraction (commonly called PUREX) process to separate the uranium and plutonium. The plutonium fraction was transferred to the War Reserve plutonium recovery stream. The uranium fraction was precipitated with ammonia gas and calcined to uranium oxide. If the plutonium was <500 ppm, this oxide was shipped to the Idaho Chemical Processing Plant (now Idaho Nuclear Technology and Engineering Center) at INL.

12.7 Oralloy-Contaminated Equipment

Building 889 was constructed as a decontamination facility for DU- and HEU-contaminated items that could be used elsewhere in the plant. However, a significant amount of Oralloy-contaminated equipment was identified as surplus and was crated and shipped to INL (see Appendix DD). The chemical constituents in the bath salts were sodium, potassium, and lithium carbonates, which formed a eutectic salt bath. A property disposal record form is employed to write-off capital items. The D83 numbers are capital equipment numbers and not drum numbers in this case.

12.8 1969 Fire Waste

On May 9, 1969, RFP experienced a disastrous fire in the plutonium foundry, Building 776. The fire cleanup generated a significant quantity of plutonium-contaminated waste. For costing information and to differentiate fire recovery waste from routine-generated waste, a special identification number of (771-596) was used. Special numbers (A00----) were assigned to each crate shipped to INL. See Appendix EE for a list of the crated fire waste shipped, which covers shipments over a period from October 1969 to February 1972. Included in Appendix FF is a copy of the procedure employed to determine the amount of plutonium on equipment and machine tools and a copy of a letter addressing the limitations associated with wooden waste shipping crates in ATMX-600 railcars.

12.9 903 Pad Disposal

The generation of machine cutting oil and other organic solvents grew into a storage and processing dilemma. This situation forced outside storage on the 903 Pad. A treatment process was finally developed in 1966 that converted the organics into a grease and was identified as Series 743 sludge. Processing the estimated 5,230 drums began in January 1967. The chronology of processing is shown in Table 20 with a summary provided in Table 21.

Table 20. 903 Pad drum processing chronology.

Time Schedule—Processing Lathe Coolant Solutions (from M. Maas monthly progress reports)	
1/23/67	Planning started for processing lathe coolant solutions.
3/67	Start of 2-month trial period for processing lathe coolant. 191 drums processed during trial run.
5/67	Decision made to process drums of coolant from 903 storage area. Estimate 5,230 drums of organic liquid in 903 storage area.
11/27/67	Emptying of drums in the field is 70% complete.
1/25/68	3,860 drums removed to date, 77% complete.
2/27/68	Approximately 1,250 (24%) empty drums removed to date.
3/29/68	4,146 (78%) drums of organic waste removed to date.
4/26/68	2,614 (53%) empty drums removed from 903 storage area to date.
5/21/68	Last pumpable contaminated organic liquid removed from 903 storage area.
5/28/68	Final 41 drums of unpumpable greases and tars removed from 903 storage area.
6/7/68	Completed removal of all drums and pallets. Final policing of 903 storage area complete.
9/24/68	Final report on removal of all drums from 903 storage area.

Table 21. Disposal summary of plutonium-contaminated materials, 903 Pad storage area.

Total drums at start	5,230 drums
Drums sent to Building 774 for processing	4,826 drums
Drums containing plutonium contamination	3,572 drums
Empty drums (includes original empty drums plus drums emptied after solution removal)	4,672 drums ^a
Total drums processed in Building 774 with an average of 1.7 g of plutonium per drum	4,826 drums

a. All 4,672 empty drums were assayed before packaging and shipping to Idaho National Laboratory.

12.10 Asbestos and Miscellaneous Waste Items

Using asbestos items was very common in the Building 444 uranium foundry. The following is a list of the asbestos items used in foundry operations and discarded to waste when contaminated with DU:

- Aprons
- Fire blankets (4 × 8 ft)
- Gloves

- Jackets
- Hoods
- Shin guards
- Tape.

These items could have been discarded as Type I combustibles, Type V noncombustibles, or both, depending on the operator's discretion.

Other sources of asbestos in all areas were HEPA filters, filter media, and furnace insulation.

A list of materials that were discarded to waste from Building 444 is provided below. These items made up a very minor quantity of the waste generated and shipped:

- Grinding wheels and motors
- Unclassified tooling
- Cadmium turnings from back machining
- Chromium plating turnings from back machining
- Lead casting residues—skull and turnings
- Aluminum chips, turnings, and casting skull
- Copper turnings and casting skull
- Spent furnace brick
- Contaminated furniture.

12.11 High-Efficiency Particulate Air Filters

Buildings handling radioactive material at RFP used various stages of HEPA filtration on the glove box and corresponding exhaust and intake systems. The spent exhaust filters became a disposal problem because of plutonium contamination levels, acid fumes, and other aerosols. Extensive R&D and pilot experiments were carried out on a continuing basis to provide the most effective and efficient filtration systems. The most significant information will be addressed in this section. See Appendix GG for a list of reports on efforts to improve the filtration systems.

12.11.1 Background

From 1953 to 1957, all plutonium production work was carried out in Buildings 771 and 991. Chemical recovery, metal recycle, foundry, and machining operations were performed in Building 771. Final assembly of plutonium components and shipping preparation was accomplished in Building 991. Very little plutonium waste was generated by Building 991. Building 774 was constructed to process the aqueous radioactive waste generated by Building 771 and the uranium buildings on plant site into sludge for shipment offsite.

In 1957, Buildings 776 and 777 were constructed to accommodate the increased plutonium mission. Foundry and machining operations were moved to Building 776, while Building 777 handled final assembly operations. A concrete block wall separated the two facilities within the same structure.

Chemical recovery and metal recycle remained in Building 771. Foundry and machining capabilities in Building 771 were taken over by the R&D group.

Production operations in Building 776 were essentially dry processes, except for the degreasing solvents and lathe coolant, which were organic-based liquids. The recovery and recycle processes in Building 771 were mainly aqueous processes using large quantities of nitric acid. Consequently, the glove-box atmospheres were much more corrosive in Building 771 than in Buildings 776 and 777, which adversely affected the HEPA filters in Building 771 compared to Buildings 776 and 777.

12.11.2 Plutonium Ventilation Systems

The ventilation filtration systems in the plutonium buildings can be segregated into two connecting units:

- From the processing glove boxes through the HEPA filters in the booster system
- From the booster system through the final HEPA filters in the final plenum and out the exhaust stack to the atmosphere.

The booster and final plenum systems have two filter banks that are called first- and second-stage HEPA filters. The glove boxes have a small intake HEPA filter and a small exhaust HEPA filter. The intake filter is discarded normally as non-TRU waste. Glove-box operations that produce minimal amounts of dust and aerosols have exhaust filters that will satisfy criteria for non-TRU waste.

12.11.3 Before 1964

Zodtner and Rogers (1964) addressed, in part, plutonium loading onto HEPA filters for Buildings 771 and 776. Plutonium loading was reported based on the two filtration units described above.

First- and second-stage HEPA filters in the booster system for Building 776 generally load to approximately 50 g of plutonium for the lifetime of the filters as determined by radiometric counting techniques. For the final plenum filters, plutonium loading for the lifetime of the filters was negligible based on radiation counting procedures.

Booster and final plenum filters were packaged and shipped offsite. Plutonium loading for the booster filters from Building 776 was estimated by Zodtner and Rogers (1964) as 25 g per filter. The reduced plutonium loading value was to compensate for filters that were removed before full plutonium loading because of filter damage and premature plugging by foreign material.

First- and second-stage HEPA filters in the Building 771 booster system became highly contaminated with plutonium attributed to the chemical processing involved in plutonium recovery. Plutonium loading for the booster filters has been determined to average about 300 g per filter. This estimate is based on burning a full set of booster filters and assaying the resultant ash. The estimated plutonium value for shipping these filters offsite was 200 g per filter as assigned by Zodtner and Rogers (1964). Again, the plutonium loading per filter was reduced to compensate for premature removal of some filters before reaching full loading. For first- and second-stage final plenum filters, Zodtner and Rogers assigned an estimated plutonium loading value of 2 g per filter.

Plutonium loading of HEPA filters in ventilation systems for Buildings 774 and 991 was considered to be negligible. Consequently, the HEPA filters for those buildings were considered low-specific activity waste in the terminology of that time.

The Zodtner and Rogers (1964) report estimates that 70% of the 2,358.7 MT (2,600 tons) of waste sent to INL was generated as building waste. This waste was only slightly contaminated and should not contribute heavily to the burial ground contamination. About 15% of the waste sent originated from within the glove-box lines and was the main contributor to the plutonium levels in the burial pits. The remaining 15% of the waste sent consisted of obsolete and nonrepairable equipment and bulky items too large for a 55-gal drum. These contaminated items were disposed of in large wooden waste boxes and were a minor contributor to the plutonium levels.

The two major plutonium waste generators were Buildings 771 and 776 with a minor amount coming from Building 777. A waste generation distribution is shown for Buildings 771 and 776 in Tables 22 and 23, respectively.

Waste generated by the glove-box line consisted of combustibles such as rags, paper, plastics, and rubber gloves. Noncombustibles consisted of scrap metal, broken glass, heavy rubber items, and small process equipment items. A small quantity of graphite was included with the line-generated waste.

Table 22. Waste shipments to Idaho National Laboratory from Building 771.

Calendar Year	General Building Waste		Glove-Box Line Waste		Wooden Box Waste	
	Number of Drums	Weight (ton)	Number of Drums	Weight (ton)	Number of Boxes	Weight (ton)
1954	1,036	31	40	3	5	—
1955	1,383	78	60	4	6	1
1956	1,825	103	80	5	7	1
1957	3,180	217	130	9	57	16
1958	2,143	145	90	6	122	37
1959	2,609	165	110	7	105	34
1960	2,414	173	100	7	124	69
1961	2,301	144	130	9	48	18
1962	3,426	222	110	7	103	54
1963 ^a	1,183	112	50	3	73	66
Totals	21,500	1,390	900	60	650	296

a. 6-month total.

Table 23. Waste shipments to Idaho National Laboratory from Building 776.

Calendar Year	General Building Waste		Graphite Waste		Washable Waste		Miscellaneous Waste		Wooden Box Waste	
	Number of Drums	Weight (ton)	Number of Drums	Weight (ton)	Number of Drums	Weight (ton)	Number of Drums	Weight (ton)	Number of Boxes	Weight (ton)
1957	71	2	5	1	5	1	30	2	0	0
1958	447	24	100	12	25	2	100	10	7	1
1959	753	35	150	19	50	3	200	20	27	7
1960	721	44	110	14	43	3	95	9	26	11
1961	1,092	73	451	49	60	4	188	20	36	13
1962	1,409	87	521	68	83	6	169	18	26	11
1963 ^a	777	48	0	0	0	0	51	5	3	4
Totals	5,270	313	1,337	163	266	19	833	84	125	47

a. 6-month total.

The contents of the boxed waste consisted of spent process equipment, duct work, and piping, which had many inaccessible areas that could retain substantial amounts of plutonium. Sections of duct work and piping, when removed, were immediately sealed and placed in a box without cleaning. At that time, no reliable or practical method of measuring the plutonium was available. Consequently, the plutonium shipped offsite in these boxes could vary from contamination count levels to hundreds of grams.

Waste-generating streams in Building 776 can be segregated into five categories listed below:

1. General building waste
2. Graphite waste
3. Washable waste
4. Miscellaneous waste
5. Boxed waste.

General building waste was similar to that for Building 771 and was considered to be slightly contaminated with insignificant amounts of plutonium.

Graphite waste was generated by the plutonium foundry through casting procedures. Graphite molds were probably the largest contributor to plutonium sent to INL in the early years. Building 776 washable waste consisted of rubber-and plastic-based materials. These materials received a washing before being sent offsite. The miscellaneous waste generated in Building 776 was given a superficial cleaning before being placed in 55-gal drums for shipment offsite.

12.11.4 Uranium Processing Building

Building 881, which started operation in 1953, manufactured HEU (93% U-235) components coupled with a chemical recovery and metal recycle capability. Manufacturing HEU ceased in 1964, but chemical recycle and other chemical functions continued into the early 1970s. The HEPA filters were sent

offsite also. No records were located indicating the amount of HEU shipped offsite with these filters in the 1950s and 1960s.

Building 444, which started operation in 1953, manufactured DU components and, later, beryllium components. The HEPA filters from Building 444 and later from Building 447 were sent offsite also. The amount of DU contamination for the HEPA filters was not recorded.

Building 883, which started operation in 1957, rolled and formed DU and HEU. The HEPA filters from Building 883 were sent offsite also. The amounts of DU and HEU on the filters from Building 883 were not recorded but treated as low-specific activity waste for offsite shipment.

The CWS filters in the HEU area located in Building 881 became loaded with ammonium nitrate salt. The formation of ammonium nitrate salt on the plenum filters was attributed to using nitrate acid and ammonia (gas) in HEU chemical recovery processes. To extend the life of these filters, the plenum filters were subjected to steam humidification to dissolve ammonium nitrate salt, thereby permitting air passage (see Appendix HH). Consequently, traces of ammonium nitrate may be present on CWS filters from Building 881.

12.11.5 High-Efficiency Particulate Air Filter Plutonium Measurements

Before 1964, the plutonium on the HEPA filters was not measured and was not taken as a measured discard. Zodtner and Rogers (1964) initiated an effort to measure the plutonium on discarded HEPA filters and to develop recovery procedures for filters highly loaded with plutonium. Economic discard limits were developed in the late 1960s for HEPA filters. As indicated in Table 14, the economic discard limit for glove-box filters was 3 g per filter, and the economic discard limit for the larger plenum filters was 24 g per filter. The discard limits for filter media in 1992 are listed in Table 24.

Table 24. Plutonium discard limits for filter media in 1992.

Filter Media	Discard Limits (g/g)
Item Description Code 338 high-efficiency particulate air filter	0.007380
Item Description Code 335 absolute drybox filter	0.006000
Interstate Commerce Commission 376 processed filter	0.007380

The initial economic discard limits were probably in the range of the 1992 limits.

A program to develop NDA methods for drummed and boxed waste was initiated in 1963. The first production drum counter was activated in 1969 followed by several upgrades in the early 1970s; however, NDA methods were developed in the mid-1960s and employed on a pilot scale for verification studies.

A plutonium recovery procedure was developed for processing “wet” HEPA filters and “dry” HEPA filters exceeding the economic discard limit. The procedure was implemented in the mid-1960s. The filter was removed from its frame, leached with dilute nitric acid, washed with water, and subsequently dried. The wooden frames were shipped as low-specific activity waste or burned in the Building 771 incinerator if highly contaminated with plutonium.

12.12 High-Efficiency Particulate Air Filter Management

Buildings at RFP that handled radioactive materials were constructed with appropriate intake and exhaust air filtration ventilation systems. The air exhaust systems were the main concern as these systems provided the environmental protection against the release of radioactive particulates to the surrounding atmosphere. The spent exhaust filters were shipped to INL from 1954 through 1970 and designated as Type III CWS filter waste.

An internal report by Zodtner and Rogers (1964) has raised concern at INL over the large amounts of plutonium arbitrarily assigned to exhaust filters from the plutonium processing buildings. At that time, the two buildings of concern were Buildings 771 and 776, which processed and handled large quantities of plutonium-bearing materials. Consequently, this section will omit discussing the uranium processing buildings and concentrate mainly on Buildings 771 and 776. The other buildings, which came in contact with plutonium, did not have accountable amounts of plutonium on their exhaust filters.

12.12.1 Plutonium Exhaust Ventilation System

The exhaust ventilation system used in the plutonium buildings consisted of three filtration components based on the filters employed: (1) glove-box filters, (2) booster plenum filters, and (3) main building plenum filters. The air entered the glove box through an intake filter and exhausted to the booster plenum through a glove-box exhaust filter. The glove-box exhaust air transferred through the booster plenum to the main building plenum. The air passed through the main plenum and exhausted to the building stack.

The booster plenums contained two or four filter stages, while the main plenum originally had two filter stages. All booster plenums and main building plenums after 1970 were upgraded to four stages of filters coupled with automatic sprinkling systems for fire suppression. A simplified diagram of the three components and the exhaust air routing is shown in Figure 7. Not shown in Figure 7 is the exhausting of room air to the main building plenum.

The production operations in Buildings 776 and 777 were essentially dry processes except for the degreasing solvents and lathe coolants, which were organic-based liquids. The recovery and recycle processes in Building 771 were mainly aqueous processes employing large quantities of nitric acid. Consequently, the glove-box atmospheres were much more corrosive in Building 771 than in Buildings 776 and 777, which adversely affected the HEPA filters in Building 771 compared to Buildings 776 and 777.

12.12.2 Filter Background

The filters used for air filtration exhaust systems varied as necessary to improve particulate removal from exhausted air to the surrounding environment. The initial CWS filter employed impregnated paper for the filter media. The 1957 fire in Building 771 burned the paper CWS filters, which called for a fire resistant filter media. Consequently, a glass-asbestos filter was introduced in the 1959–1960 timeframe. This timeframe is somewhat substantiated by the low number of filters shipped in 1960 and the high number of filters shipped in 1959 as reported in Table 25. This shipping event indicates the possibility of a filter changeout.

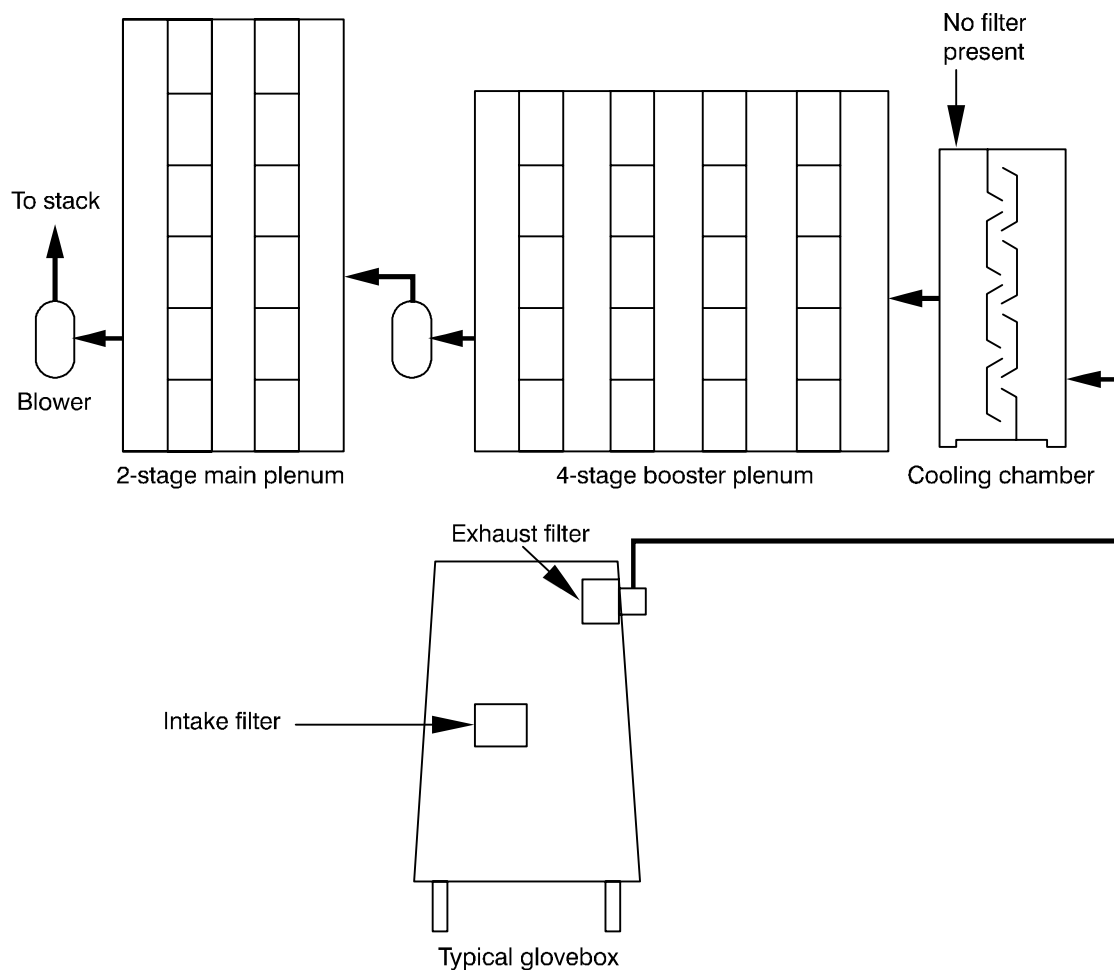


Figure 7. Plutonium ventilation system.

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The carcinogenic nature of asbestos required the development of filter media that were fire resistant and eliminated the carcinogenic factor. Consequently, R&D efforts to coordinate with filter manufacturers were accelerated in the 1970s and into the 1980s. The RFP historical records provide a significant amount of documentation on studies of HEPA filter media, filter constructing, filter testing, and associated waste reduction efforts. However, this copious documentation was not directly relevant to the 1954–1970 period of interest. The present assumption is that the paper CWS filters were used up to about 1959 and were replaced with the CWS glass-asbestos filters through 1970 and beyond.

12.12.3 Filter Generation Rates

The waste identification system employed during the 1954–1970 period did not record ventilation filter generation rates by building or function. The designation used was Type III CWS filters. However, a summary report by the Waste Disposal Coordination Group listed the number of filters shipped to INL for the 1954–1970 period by year (see Table 25). Table content was obtained from monthly and annual reports of waste shipping history prepared by the Waste Disposal Coordination Group. Anderson’s internal report (see Appendix II) lists the backlog of CWS filters as 463 as of June 1966 and also places the generation rate at approximately 20 filters per month.

Table 25. Summary of filters shipped to Idaho National Laboratory taken from reports prepared by the Waste Disposal Coordination Group.

Calendar Year	Number of Filters	Number of Cartons
1954	0	0
1955	1,205	0
1956	2	0
1957	1,251	101
1958	1,042	123
1959	1,679	0
1960	130	34
1961	1,592	0
1962	549	7
1963	535	0
1964	1,023	0
1965	762	0
1966	575	10
1967	990	948
1968	323	4,267
1969	209	249
1970	641	43
Total	2,508 ^a	5,782 ^b

a. Includes 24 × 24 × 14-in., 24 × 24 × 16-in., 24 × 24 × 18-in., 24 × 24 × 28-in., and 28 × 28 × 16-in. cartons of filters

b. Includes 5,496 cartons containing 55-gal drums.

12.12.4 Filter Configuration

Historical RFP documentation addressing the configuration, construction, and materials of construction was limited at best; however, Anderson (see Appendix II) includes a description of the backlog CWS filters. The CWS Type F filter was manufactured by Flanders and Cambridge; filter dimensions were 24 × 24 × 12 in. with a weight of about 22.7 kg (50 lb). Eventually, the CWS filters were replaced with HEPA filters manufactured to AEC standards.

The notes on the bottom of Table 25 list the sizes of the cartons used to ship filters to INL. From the sizes listed, the 24 × 24 in. appears constant, while the depth varies as indicated by 14 in., 16 in., 18 in., and 28 in. An exception to the 24 × 24-in. configuration is the 28 × 28 × 16-in. carton. A reasonable assumption for the 28 × 28 × 16-in. carton and the 28-in.-deep carton is to double the number of filters per carton.

12.12.5 Filter Media

From plant startup to about 1960, the CWS filters had a cellulose base for the filter media, which were combustible. The 1957 fire in Building 771 demonstrated that the cellulose-based filter media were

unacceptable because of this combustible characteristic. Consequently, a search and development program began to provide noncombustible filter media with acceptable filtering capability.

A CWS Type F filter manufactured by Flanders and Cambridge replaced the combustible CWS filter in the 1959–1960 timeframe. The materials of construction for the CWS Type F filter are listed below as reported by Anderson (see Appendix II):

- Size: $24 \times 24 \times 11.5$ in.
- Weight: 22.7 kg (50 lb)
- Filter media: glass-asbestos
- Separator: aluminum or asbestos
- Frame: wood or cadmium-plated steel.

As stated previously, the carcinogenic nature of asbestos demanded that the Type F CWS glass-asbestos filters be phased out. Post-1970 documentation indicates that the glass-asbestos filter was employed well into the 1970s. Consequently, the filters shipped to INL from 1954 through 1960 were the cellulose-based CWS filters, followed by the glass-asbestos filters.

12.12.6 Filter Discard Limits

The plutonium material unaccounted for that became apparent in the late 1950s and early 1960s was the driving force to establish a system to determine what should be declared waste and what should be processed for plutonium recovery. A system was developed based on comparing the cost of producing a gram of reactor plutonium to the cost of recovering a gram of plutonium from residue waste. The break-even point was established as the economic discard limit for a given plutonium residue (see Section 6.6).

To establish economic discard limits for the large number of generated waste residues required developing NDA procedures for plutonium assays, instituting an appropriate cost accounting system, and staff training. The first NDA drum counter was developed in 1964, which provided estimates for plutonium content in waste residue. The first production drum counter was placed into use in 1969. The economic discard limit program became effective in the late 1960s.

For most of the plutonium-bearing waste residue, the economic discard limits were established on a gram of plutonium per gram of the matrix material. Filters were the exception, as the economic discard limit was set per filter rather than a weight-to-weight ratio. The discard limit for filters is reported by Anderson, Putzier, and Ziegler (see Appendix I); limits shown in Table 26 are for 1968.

Table 26. Filter economic discard limits for 1968.

Filter Media	Dimensions	Discard Limit
Chemical Warfare Service filter	$2 \times 2 \times 1$ ft	24 g/filter
Drybox filter	$8 \times 8 \times 4$ in.	3 g/filter

12.12.7 Plutonium Filter Content

The pre-1970 historical documentation does not address the levels of plutonium contamination on spent glove-box and plenum filters other than the estimates provided by Zodtner and Rogers (1964). Terada, Woodard, and Jensen (1985) provide plutonium levels for plenum filters. For 30 filters located in the FU-2B booster plenum in Building 771, the range of plutonium content varied from 6 g per filter to a high of 46 g per filter.

Anderson (see Appendix II) estimates that the backlog of 463 filters contains 34.6 kg (76.3 lb) of plutonium. The average plutonium content per filter would be 75 g. An additional estimate for generation placed at 20 filters per month was 300–1,000 g of plutonium. The average plutonium content per filter would range between 15–50 g of plutonium. The accumulated backlog of filters cited by Anderson indicates that highly contaminated filters were not being shipped in the mid-1960s.

12.12.8 Plutonium Recovery from Spent Filters

The plutonium recovery process described by Anderson (see Appendix II) includes a water rinse to remove any residual nitric acid. Briefly, the filter media were removed from the wood or metal frame and leached with concentrated nitric acid. Calcium fluoride was added to the leach solution as a source of fluoride ion to accelerate the dissolution of plutonium oxide. The leach solution was transferred to anion exchange processing to purify and concentrate the plutonium for further purification. The leached filter media were packaged for shipment into 55-gal drums with magnesia cement added for liquid absorption. The wood and metal frames were considered low-specific activity waste in the terminology of the time. In the 1970s, filter media processing became more sophisticated as evidenced by James (1980).

12.12.9 Filter Processing

The HEPA filters from process glove boxes and filter plenums comprise another category of residue, which required preprocessing before plutonium recovery. These filters were processed in Line 48 using saws, hammers, and screwdrivers to separate the wooden frames from the filter media. The wood was normally discardable; the filter media, if above discard, could be transferred to Line 21B for further processing. In Line 21B, the filter media were first contacted in a closed reactor vessel with anhydrous hydrofluoric acid that removed silica from the matrix. The plutonium-bearing matrix was then immersed in concentrated nitric acid in a batch dissolver. The resulting plutonium nitrate solution was filtered and transferred by vacuum to ion exchange. The remaining solids were reprocessed, if above the discard limit, or shipped to waste disposal if below the discard limit.

Glove-box filters were removed from their holding frame and tapped inside a stainless steel tray to remove any loose particulate matter. Based on visual examination and processing history for a given glove box, the operator or foreman made a decision to process or ship as waste. This procedure continued until NDA systems became available in the late 1960s. The history of the glove-box processing was important as large quantities of dust were generated in grinding, scraping, and hammer milling. In other glove boxes, the operations performed were dustless and dry, which produced very little plutonium contamination on the glove-box exhaust filter.

Certain booster plenums served processes that generated acid fumes and dusts, which contaminated the booster filters. Consequently, these booster filters were changed more frequently and were more likely to be processed for plutonium recovery.

12.12.10 Waste Filter Packaging

Spent plenum filters, which were considered waste, were packaged into cartons for shipment to INL. Those plenum filters considered to have recoverable amounts of plutonium were sent to plutonium recovery for processing. This visual examination process continued until NDA systems were developed in the mid-1960s to assay the spent filters for plutonium content.

Glove-box filters were removed from the glove-box line through a bag-out procedure. Often a double-bag procedure was followed. Bagged filters that were considered recoverable were transferred to the leaching glove-box line. filters were placed in a 55-gal drum lined with a poly liner. The operator or the foreman estimated the plutonium content. This estimate was included in the normal operating loss for a given material balance area account.

About 1967 or 1968, economic discard limits were established, which provided a nondiscretionary procedure for determining whether a given filter was waste or recoverable.

The waste-generating streams in Building 776 can be segregated into five categories, which are listed below:

- General building waste
- Graphite waste
- Washable waste
- Miscellaneous waste
- Boxed waste.

The general building waste was similar to that for Building 771 and was considered to be slightly contaminated with insignificant amounts of plutonium.

The graphite waste was generated by the plutonium foundry through casting procedures. The graphite molds were probably the largest contributor to the plutonium sent to INL in the early years. Building 776 washable waste consisted of rubber-and plastic-based materials. These materials were washed before being sent offsite. The miscellaneous waste generated in Building 776 was cleaned superficially before being placed into 55-gal drums for shipment offsite.

12.13 Waste Drums Returned to Rocky Flats Plant

Drum surveillance at INL identified drums with excessive plutonium levels and other problems. On several occasions, these selected drums were returned to RFP for interrogation and content inspection. Results were reported by RFP through internal reports. The inspection results varied widely and depended on each drum. Consequently, the inspection results are not addressed here, but the inspection reports are provided in Appendix JJ.

12.14 Mound Disposal

In the early 1950s, drums of DU, HEU, and low-level, plutonium-contaminated waste were buried in an area called Mound. The initial burial started in April 1954 and continued until September 1958. A total of 1,045 drums of oil and solid waste was buried. The majority of the radioactive contamination was DU with some HEU and possible low-level plutonium. The drums were exhumed with the oil drums transferred to the 930 pad. Completed retrieval and offsite disposal were accomplished by May 1970. Shipment to INL was noted on several trailer load lists.

12.15 Reactor Grade Plutonium

Rocky Flats Plant did not process any irradiated reactor fuel material. However, in the late 1960s, RFP fabricated fuel elements for the Zero Power Physics Reactor at the Materials and Fuels Complex (formerly Argonne National Laboratory-West). The fuel element alloy was composed of DU, plutonium, and molybdenum, with DU being the major component. The plutonium material had a Pu-240 content in the 8–10% range.

The residues and waste generated were segregated from War Reserve production and were shipped to Hanford. No packaged residue or waste was shipped to INL. However, trace amounts of Zero Power Physics Reactor material were probably commingled in the organic liquid waste and the aqueous waste sent to Building 774.

12.16 Cyanide Waste

Cyanide salts were used in Building 444 in heat-treating baths. Disposal of these spent baths was a waste disposal problem as indicated by Ryan (see Appendix KK):

The disposal of cyanide wastes which are produced in Building 44 are a potential problem. In the past, these wastes were set-up with Portland cement in Building 44. At the present time, these wastes are being sent to Building 81 for destruction. The presence of fluoride and Building 44 material in the waste makes this method undesirable for Building 81.

Because of potential safety concerns and disposal problems, cyanide heat-treating baths were eliminated in favor of carbonate-based baths.

12.17 Glove-Box Gloves

The plutonium-handling buildings at RFP used significant quantities of glove-box gloves coupled with an inspection program to detect failing gloves. Consequently, defective gloves contaminated with plutonium were shipped to INL. Giebel and Riegel (1971) provide specifications for glove-box gloves for procurement purposes. The report identifies glove materials, design parameters, and requirements.

12.18 Liquid Organic Waste

Manufacturing operations in the uranium and plutonium areas used a variety of organic liquids that became a salvage and disposal problem in the 1950s and early 1960s. Installation of a solidification process in the middle 1960s in Building 774 (known as the Grease Plant) produced disposable sludge from these organic liquids.

These organic liquids were used for machine lubricants, coolants, cutting oils, vacuum and diffusion pump oils, hydraulic oils, and a variety of degreasing and cleaning solvents. Monobromo benzene was initially used in determining the density of plutonium components. To eliminate potential health and flammability hazards associated with monobromo benzene, Freon 113 was substituted.

In the late 1950s and early 1960s, aqueous-based coolants and lubricants were substituted for organic-based oils in the uranium areas. This substitution reduced the potential fire hazard associated with pyrophoric uranium turnings and organic oils.

A variety of chlorinated hydrocarbon solvents were used based on their toxicity and their compatibility with product and equipment. The most significant solvent and lathe coolant diluent was CCl₄. Significant data from Hobbs (1982) regarding usage of CCl₄ at RFP are listed below:

- Review of purchasing data indicated about 60,566.6 L (16,000 gal) per year of CCl₄ used in the 707 plutonium manufacturing facility
- A total of 41,639.5 L (11,000 gal) of CCl₄-oil mixture sent to waste in Fiscal Year 1981
- Waste stream estimated to contain an average of 70 vol% CCl₄
- Estimated about 136.1 kg (300 lb)/day CCl₄ lost to evaporation during usage.

Hawes (see Appendix LL) reported the usage of 52,995.8 L (14,000 gal) of CCl₄ over a 12-month period. These two reports indicate a very large usage of CCl₄ per year of which about 50–70% ended up in the Grease Plant Series 743 sludge.

The total purchase of TCE for Buildings 707 and 777 was 9,274.3 L (2,450 gal) in 1989. The amount of Freon 113 used in Building 707 ranged from 3,028.3 to 3,406.9 L (800 to 900 gal) per year. Building 777 switched from using isopropyl alcohol to TCE for cleaning activities in the 1963–1964 timeframe. At this time, PCE was replaced with TCE.

In 1958, a cutting oil was used in the plutonium machining operations to facilitate machining and to reduce spontaneous combustion of the plutonium turnings. Shell Vitera cutting oil was initially used followed by a PCE washing. Later, PCE was replaced with CCl₄ as PCE attacked the glove-box gloves. Later, Shell Vitera was replaced by Texaco Regal Oil, which costs less. This replacement was in the 1970–1972 timeframe. In general, the oils used in the plutonium metal working areas were paraffin-based mineral oils with ~0.5 wt% of an antioxidant additive.

As stated previously, accumulation of this organic liquid waste was a significant disposal problem as indicated by volumes shown in Table 27 for a period in 1962. The majority of waste organic liquids was contaminated with DU, HEU, or plutonium.

Table 27. Organic waste.

Waste	Source (Building No.)	Disposition	Rate of Accumulation ^a (gal/month)
Machine coolant (Shell Vitera-CCl ₄)	776	200,000 gal in storage	4,500
Distillation still bottom	444	2,000 gal in storage (some buried in open pit)	200
Distillation still bottom (chlorinated)	444	3,500 gal in storage	50
Machine coolant	881	Buried in open pit	500
Chlorinated solvents	881	3,500 gal in storage	50
Cold oils	Miscellaneous	Buried in open pit	1,000
Trichloroethylene	777	400 gal in storage	90
Trichloroethylene	991	Will be stored	180
Miscellaneous organics	777, 771, 444	Accumulated	70

^a Rates for May, June, and July 1962.

12.19 Sludge

Sludge inevitably formed wherever liquids were used in operations and facility systems. Sludge in general can be characterized as follows:

- Organic-based
- Acidic-based
- Caustic-based
- Organic-aqueous-based.

Sludge formed in process equipment such as dissolvers, leaching vessels, degreasing vats, storage tanks, pumps, distillation bottoms, gear boxes, hydraulic presses, quenching tanks, and heat-treating baths.

12.19.1 Organic-Based Sludge

Typical organic sludge types and their origin are listed below:

- Machining coolant
- Cutting oils
- Vacuum pump oils
- Degreasing solvents

- Hydraulic fluids
- Lubricating greases and oils.

The radioactive contamination in this sludge was particulate matter. In general, contamination was low and below economic discard limits.

The disposition of organic sludge depended on its viscosity, consistency, and the availability of disposal avenues. Based on the discretion of operating personnel and waste management concurrence, organic sludge could be shipped to INL, disposed of on plant site, or stored. The installation of the Grease Plant in 1966 ultimately became the disposal route for much of the stored organic sludge. On the load lists, non-Building-774 sludge was often identified as Type IV sludge with or without the Series 743 designation.

12.19.2 Acidic-Based Sludge

Acidic-based sludge was formed in process equipment such as dissolvers, evaporators, pumps, and tanks. This sludge was characterized by its acidity and plutonium content as indicated below:

- High nitric acid with high plutonium content
- High nitric acid with low plutonium content
- Low nitric acid with low plutonium content
- Low hydrochloric acid with low plutonium content and chloride salts present.

The dissolution of this sludge produced solutions that were either sent to plutonium recovery or transferred to Building 774 based on the plutonium concentration. If sent to Building 774, the sludge solutions would become Series 741 and 742 sludge for shipment to INL or would be deposited in the solar evaporation ponds.

12.19.3 Caustic-Based Sludge

Caustic-based sludge was characterized as indicated below:

- High caustic (NaOH/KOH) with low plutonium
- Low caustic (NaOH/KOH) with low plutonium.

This sludge was usually discardable as the plutonium content was very low and in the form of particulates. The sludge was dissolved by the addition of water or low nitric acid, filtered, and transferred to Building 774 for final treatment.

12.19.4 Organic-Aqueous-Based Sludge

The organic aqueous-based sludge formed mainly in the uranium-plutonium recovery process, which employed a TBP-dodecane extractant. This sludge was handled by the Special Recovery Group to limit the introduction of organic material to the Building 774 aqueous stream and to control the quantity of aqueous solutions to the Grease Plant.

12.20 Mercury Waste

Mercury was used at RFP in diffusion vacuum pumps, instrumentation, and analytical laboratory procedures. The analytical laboratory recycled its mercury for reuse using a triple distillation procedure. The distillation bottoms were bottled and transferred to Building 774 for disposal. Other sources of spent mercury were transferred also to Building 774. The bottled mercury was discarded in a drum of solidified aqueous sludge based on operator discretion.

12.21 Excess Chemical Compounds

Excess chemical compounds accumulated and required a disposal route. Unopened containers were provided to local universities, colleges, schools, and other governmental agencies. Excess noncontaminated chemical compounds that were water soluble were added to the solar evaporation ponds next to Building 774, provided the compounds were compatible with solar pond constituents. These chemical compounds ended up in the Series 745 evaporation salts processed through Building 774 and sent to INL.

Chemical compounds not acceptable in the solar ponds were treated by the generator to fit a waste stream or sent to Building 774 for disposal. Building 774 accommodated these chemical compounds through the Series 744 sludge process or spoon feed to an acceptable waste treatment stream that produced Series 741, 742, and 743 sludge. The disposition route was governed mainly by the quantity received for disposal.

12.22 Polychlorinated Biphenyls

Polychlorinated biphenyls were used throughout RFP in electrical transformers, capacitors, hydraulic presses, and vacuum diffusion pumps. During the 1954–1970 timeframe, the polychlorinated biphenyls shipped to INL probably came through combustible waste. Leaks from hydraulic and other equipment were taken up using rags and absorbent wipes, which were sent to INL if generated in DU, HEU, and plutonium areas. Polychlorinated biphenyls were phased out in the 1970s.

12.23 Complexing Agents

Complexing agents were used by the analytical laboratories and as a constituent in decontamination solutions. The amount of complexing agents used by the analytical laboratories was minor when compared to the quantity used in decontamination efforts.

The analytical lab solutions with complexing agents were collected and treated for disposal by the generating laboratories or by chemical recovery in Building 771. If neither the laboratories nor chemical recovery could dispose of complex aqueous waste, the complexing waste was bottled and sent to Building 774. These solutions were treated by cementation techniques for disposal at INL.

The contaminated decontamination solutions were collected and transferred to Building 774. These solutions were cemented for disposal at INL. At the discretion of Building 774 operators, bottles of hot decontamination solutions would be included with Series 742 and 744 sludge.

12.24 Analytical Methods

The INL personnel have been concerned about the accuracy of plutonium and Am-241 determinations in waste sent to INL before 1971.

The RFP HEU and plutonium analytical laboratories were participants in the AEC Sample Exchange Program. Consequently, RFP analytical results were monitored by AEC for accuracy within the sample exchange results; however, these analyses were on products such as metal oxide and rich nitrate solutions. No waste samples were involved in the AEC Sample Exchange Program.

The problem with solid waste analysis was obtaining a representative sample for analysis because the waste was not homogeneous. In the 1950s and somewhat in the early 1960s, estimates for plutonium in waste items were based on a by-difference approach coupled with operating experience. The increasing plutonium material unaccounted for was a primary concern, which led to the development of NDA methods and the installation of low-level plutonium recovery processing.

As stated previously, the first NDA drum counter was an experimental model placed in service by R&D in 1964. Drum verification studies were carried out by R&D for graphite waste (see Appendix V).

The demand for NDA standards enlarged the chemical standards group and their scope. Doherty and McBride (see Appendix MM) cite biases of 1–20% and variabilities ranging from 4 to 37%.

Lawless and Chanda (1970) provide an evaluation of a helix counter designed for assaying graphite and ash residues for plutonium content. Biases and precision results are reported for certified standards of graphite and ash matrices (see Appendix NN).

13. TIMELINE AND CORRESPONDENCE—WASTE MANAGEMENT

The timeline of interest for this report covers primarily 1954 to 1970. Consequently, the timeline presented below also covers primarily that period of interest.

Correspondence from Ed Vejvoda to Operable Unit 7-13/14 staff is listed below in Section 13.2.

13.1 Timeline

Date	Event
1952	INL—Original NRTS landfill, now known as the Subsurface Disposal Area, is established.
July 1952	INL—First trench opens for disposal of solid waste.
1952–1957	INL—Trenches 1 through 10 are excavated to basalt; average 1.8 m (6 ft) wide, 274.3 m (900 ft) long, and 3.7 m (13 ft) deep.
1953	INL—U.S. Atomic Energy Commission Idaho Operations Office becomes responsible for the operation of the burial grounds.
1953	RFP—Operations begin in Buildings 444, 771, 774, 881, and 991.
1953	RFP—Waste disposal coordination group forms with E. Ryan as contact.
1953	RFP—Radioactive waste accumulates with storage becoming a problem.
1953–1967	RFP—Contaminated organic waste disposal develops into major plant issue.
April 1954	RFP—First shipment of waste to INL. Several drums leak liquids; RFP-INL establish policy of no liquid shipments.
1954–1957	INL—TRU ^a -contaminated waste from RFP, packaged in drums or wooden crates, is stacked horizontally in pits and trenches with NRTS mixed fission product waste. Therefore, Trenches 1 through 10 ^b and Pit 1 contain NRTS waste interspersed with TRU-contaminated waste. Records from RFP do not accompany these shipments. Instead, an annual summary of disposals provided total radionuclide content and waste volume.
1954–1965	INL—Informal forms are used, no form number or revisions noted.
March 1955	RFP—First shipment of Chemical Warfare Service filters.
1956	RFP—Building 447 constructed attached to Building 444. Depleted uranium chip roaster placed in service. Building 447 houses waste management services.
1957	INL—Size of Radioactive Waste Management Complex expands from 5.3 ha (13 acres) to 35.6 ha (88 acres).
September 1957	INL—TRU waste buried in Pit 1.
November 1957	INL—Volume of waste from RFP increases rapidly, including items too large and bulky for trenches; pit disposal begins for TRU waste.
1957	RFP—Beryllium operation initiated in Building 444.
1957–1958	RFP—Buildings 776 and 777 begin operations. Plutonium foundry and machining transferred from Building 771.
1957–1971	RFP—Offsite waste shipments to INL.
1957	RFP—Assembly operations in Building 991 are curtailed and transferred to Building 777.
1957	RFP—Building 774 is designated to collect plutonium-contaminated organic liquid waste.

Date	Event
1957	RFP—Am-241 recovery is initiated for shipment to Oak Ridge National Laboratory Isotope Pool.
1957	RFP—Building 883 is constructed for HEU and depleted uranium rolling and forming.
September 1957	RFP—Building 771 fire curtails operations. Fire waste shipped to INL.
1957–1969	RFP—Building 776 is major user of carbon tetrachloride, while Building 777 is major user of trichloroethylene.
1958	INL—Landfill expands to 36 ha (88 acres).
1958	RFP—Building 771 resumes operations.
October 1959	INL—Pit 2 open; drums stacked in rows.
1959	INL—Procedures to accept waste standardized, including completion of disposal form.
1959–1961	RFP—Chip roaster is inoperative because of relocation within Building 444.
1960	RFP—Tributyl phosphate solvent extraction plutonium recovery process is replaced by anion exchange process.
1960	RFP—Chemical-Warfare-Service-treated cellulose media are replaced by glass-asbestos media for fire safety.
1960–1962	RFP—Line item is approved to expand Building 771 recovery capability and capacity.
1960–1963	(1) INL—NRTS accepts approved shipments of solid radioactive waste from offsite generators and continues accepting from RFP after commercial sites opened in 1963. (2) INL—Trenches 16 through 25 and Pits 2 through 5 open for disposal of waste and receive some mixture of RFP TRU-contaminated waste, NRTS waste, and offsite waste that is stacked or dumped.
December 1961	INL—Pit 3 opens. TRU and non-TRU waste is buried intermixed. Waste is stacked in rows.
1962–1965	RFP—Start of HEU cleanout in Buildings 881 and 883.
February 1962	INL—Pit 2 floods and disposal operations are moved to Pit 3 until September 1962.
July 1962	INL—Pit 3 waste is no longer stacked in rows; it's dumped at random until pit closure in January 1963.
1962	RFP—Beryllium sheet rolling begins in Building 883.
1962–1975	RFP—Beryllium wrought process implemented to recycle beryllium scrap.
January 1963	INL—Pit 4 opens for mixed low-level waste and TRU waste for 1.5 years and then is used for TRU waste only.
November 1963–1969	INL—Drums from RFP were dumped into pits instead of stacking.
1963–1964	RFP—Building 777 switches from isopropyl alcohol to trichloroethylene for cleaning parts.
1963–1964	RFP—Expansion of Building 771 chemical recovery facilities is completed.
January 1964	INL—In Pit 4, TRU drums are stacked in rows, and boxes are stacked along pit sides.
December 1964–July 1966	INL—Pit 4 closes; it reopens until final closure in September 1967.
1964–1969	INL—Environmental monitoring program at Subsurface Disposal Area is revised: 18 thermoluminescent dosimeters replace 35 perimeter film badges, collection and analysis of water samples from subsurface monitoring holes, and field investigations assess leaching.

Date	Event
1964–1970	(1) INL—Modifications to trenches: increase minimum depth to 1.5 m (5 ft), line bottom of excavations with at least 0.6 m (2 ft) of soil underburden, compact waste by dropping heavy steel plate on dumped waste in trenches, and increase soil cover over each disposal area to 0.9 m (3 ft). (2) INL—Trenches 33 through 49 are active.
1964	INL—In Pit 4, random dumping of waste begins.
1964	RFP—First research and development experimental nondestructive assay drum counter is established for waste assay.
1964–1966	RFP—HEU component manufacturing terminates and relocates to Y-12 plant at Oak Ridge National Laboratory.
January 1964	RFP—Zodtner and Rodgers (1964) report issued addressing plutonium material unaccounted for and possible understatement of plutonium in waste sent to INL.
February 1965	INL—Pit 5 opens for TRU waste only, apparently random placement of waste.
1965	RFP—Steam evaporator is installed in Building 774 to reduce liquid volumes stored in solar evaporation ponds. Produced Series 745 evaporator salts.
1965–1988	RFP—Initiation of Np-237 tracer program.
1966	INL—Pit 4 reopens; waste Form ID-110A first used.
1966	RFP—Series 742 and 744 sludge from Building 774 begins using 17C drums instead of 17H drums to take advantage of extra weight permitted.
May 1967	INL—Pit 6 opens for TRU waste only; boxes and drums generally segregated.
1967	INL—Pit 4 final closure.
1967	RFP—Molten salt extraction process for Am-241 removal from returned pits established in Building 776.
1967	RFP—Expansion of plutonium analysis laboratory by construction of Building 559.
1967	RFP—Concept of economic discard limits initiated for HEU and plutonium-bearing materials.
1967	RFP—Start using cardboard cartons for shipping high-efficiency particulate air filters.
1967	RFP—U.S. Department of Transportation shipping regulations require using specification 17H and 17C drums for shipping radioactive waste. Additional use of stronger drum liners and covers initiated.
1967	RFP—Grease Plant installed in Building 774 to process organic liquids in storage. Operations begins to process contaminated organic liquid stored on the 903 Pad.
1968	INL—Transition from waste Form ID-110-A to Form ID-125.
May 1968	INL—Pit 9 opens; drums are dumped.
August 1968	INL—Pit 10 opens; containers are dumped; fire waste, drums, and boxes are not segregated.
1968	RFP—Processing of contaminated organic liquids stored on 903 Pad completed with final shipment to INL.
1969	INL—Pits 9 and 10 flood.
1969	RFP—First production drum counter installed in Building 771.
1969	RFP—Approval to employ ATMX-600 railcars to haul waste to INL.

Date	Event
May 1969	RFP—Disastrous fire in Building 776 terminates operations in Buildings 776 and 777.
1969–1971	RFP—Cleanup of 1969 fire damage is completed with significant quantities of waste sent to INL over this period.
1969	RFP—Waste operations start in Building 776 and continue until closure.
January 1970	INL—In Pit 10, last drums are dumped; remainder of pit (about last 350 ft of the east end) is filled with boxes.
1970	(1) U.S. Atomic Energy Commission—New policy requires solid TRU waste to be segregated and stored retrievably. (2) INL—Burial of waste classified as TRU discontinues; TRU waste transferred to Transuranic Storage Area for retrievable storage.
April 1970	INL—Pit 11 ^c opens; drums are stacked in rows, and boxes are stacked along south wall of pit.
July 1970	INL—Pit 12 opens for TRU waste that was stacked.
October 1970	INL—Pit 11 closes.
November 1970	INL—All TRU waste is placed in aboveground, retrievable storage.
1970	RFP—Building 707 begins plutonium operations.
1970	RFP—Concept of TRU (retrievable) and low-level waste (nonretrievable) designations issued by U.S. Department of Energy.
1971	INL—Transition from waste Form ID-125 to Form ID-135.
1971	RFP—Drum counting facility constructed between Buildings 771 and 774 and designated as 771C.
1971	INL—Waste Management, as an organization, is formed and takes responsibility from the U.S. Atomic Energy Commission for disposal of radioactive waste.

a. In 1954, TRU waste was defined as TRU radionuclides in concentrations greater than or equal to 10 nCi/g

b. Pits 7 and 8 did not receive TRU waste

c. Drums in Pits 11 and 12 were retrieved 1974 through 1978.

HEU = highly enriched uranium

INL = Idaho National Laboratory

NRTS = National Reactor Testing Station

RFP = Rocky Flats Plant

TRU = transuranic

13.2 Correspondence from Edward Vejvoda to Operable Unit 7-13/14 Staff

Date	Recipient	Subject
December 4, 2001	Marianne Little	Rocky Flats Waste Information
December 11, 2001	Marianne Little	Rocky Flats Reports
December 18, 2001	Wendell Jolly	Video Tape Review
July 13, 2000	Bruce Becker	Distribution of Reference Material
December 22, 2000	Rod Thomas	Inventory Difference Briefing (3/7/84)
February 12, 2001	Rod Thomas	NDA Reports
February 16, 2001	Rod Thomas	Additional NDA System Information (1966-1977)
August 16, 2001	Bruce Becker	Your FAX July 23 2001
August 29, 2001	Bruce Becker	Plutonium Estimates for Rocky Flats Waste Forms
September 26, 2001	Bruce Becker	Nuclear Safety - Waste Management
January 28, 2002	Marianne Little	1964 Rocky Flats Drum Counter
February 12, 2002	K. Jean Holdren	EPA Region 10 Requests
March 4, 2002	Wendell Jolly	Videotape Cassette Review
April 29, 2002	Marianne Little	Rocky Flats Waste Information
July 31, 2002	Marianne Little	Rocky Flats Waste Report
August 15, 2002	K. Jean Holdren	Request for Rocky Flats Reports by DOE-IDO
November 7, 2002	Paul Sentieri	Your E-mail of November 6 2002
March 6, 2003	K. Jean Holdren	DRAFT Consolidated Report of Rocky Flats Wastes Shipped to INEEL
April 29, 2003	Marianne Little	Rocky Flats Sewage Sludge
June 3, 2003	Marianne Little	Rocky Flats Reports
June 13, 2003	Marianne Little	Returned RFP Waste from INEEL 1971
August 11, 2003	K. Jean Holdren	Distribution of Reference Material
August 25, 2003	K. Jean Holdren	Distribution of Reference Material
September 24, 2003	Marianne Little	Rocky Flats Shipments
September 30, 2003	K. Jean Holdren	AEC Courier Receipts - 1964
March 22, 2004	K. Jean Holdren	Photographs of Retrieved RFETS Waste
March 30, 2004	K. Jean Holdren	Draft Copy of Graphite Mold Report
March 30, 2004	K. Jean Holdren	Roaster Oxide Information
April 29, 2004	K. Jean Holdren	Draft Copy of HEPA Filter Report
May 20, 2004	K. Jean Holdren	Ingot Mold Drawing
June 29, 2004	K. Jean Holdren	Rocky Flats Waste Reports
July 26, 2004	K. Jean Holdren	Rocky Flats HEPA and Graphite Processing Reports
October 25, 2004	K. Jean Holdren	Transshipped Waste

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- 49 CFR 178, 2005, “Specifications for Packagings,” *Code of Federal Regulations*, Office of the Federal Register.
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Appendix A

Letter from G. V. Beard to John Epp

INTEROFFICE CORRESPONDENCE

date January 22, 1980
to J. D. McKinney
from T. L. Clements, Jr. *T. L. Clements, Jr.*
subject NON-RADIOLOGICAL HAZARDS STUDY - TLC-2-80

TYPE: Visitation - Colorado School of Mines Research Institute

File No.: RWMC-17-80

SUMMARY

A visit to the Colorado School of Mines Research Institute (CSMRI) in Golden, Colorado was made on January 15, 16, 1980. The purpose of the visit was to review available waste shipment records and interview knowledgeable personnel about waste shipments and research projects conducted during 1960-1962. Radioactive waste disposal records at the CSMRI indicate four (4) waste shipments were made to the INEL during the period of December, 1960 to October, 1962. This concurs with available INEL waste shipment disposal requests. Additional information located at the CSMRI indicates plutonium contaminated waste from a classified project was sent to the Rocky Flats Plant. Ultimate disposal of this material would have taken place at the INEL.

Available INEL Waste shipment records indicate only the October, 1962 shipment was buried in pits or trenches that may be involved in future retrieval projects. The disposal request (ID-137) identified a CSMRI purchase order. The purchase order was recovered in their archives. The purchase order identified two project numbers that absorbed the cost for disposal services. Both project files were recovered from the CSMRI archives.

Project 320311 was conducted for American Metal Climax, Inc. of Denver, Colorado. The research involved the development of rapid analytical techniques for trace elements. The identification of trace elements in ore serves as a method of determining potential ore deposits. One set of silicate rock samples and molybdenite concentrates were sent to the Argonne National Reactor in Argonne, Illinois for neutron activation. These samples were then analyzed for trace elements by various techniques, such as scintillation counting and the use of separation techniques. Chemicals used in analytical procedures would have involved acids, such as nitric, sulfuric, and hydrochloric and organic solvents such as toluene, dimethyl POPOP (1,4 bis 2,5 phenyl oxazolyl benzene), p-terphenyls, and other scintillation solutes. Complete information concerning materials used in various isotope separations and solvent extractions were not available.

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date January 22, 1980
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Information provided by F. L. Smith, past Director of Research for the CSMRI, indicated small quantities (volume is unknown) of these materials may have entered the radioactive waste stream. Chemical analysis, such as flame photometry, of unirradiated samples utilized hydrofluoric and perchloric acids. Since the samples were not radioactive, the waste chemicals from the analysis probably did not enter the radioactive waste stream.

Project 300301 was conducted for the Atomic Energy Commission, Division of Isotopes Development, under Contract No. AT(29-2)-1355, signed April 27, 1962. The title of the project was "Radioisotopes in Process Control". One aspect of the project was concerned with evaluating large volume beta and gamma detection systems for process stream analysis and automation. Initial tests for determining optimizing conditions for liquid scintillation systems utilized Cs^{137} sources. This type source allowed for comparison between liquid and plastic scintillators. A one (1) curie Co^{60} source was used to determine the effects of radiation on liquid scintillators. Waste from these projects included the radioisotope sources and general laboratory waste (paper, glassware, etc.). Scintillation solutes, such as POPOP in toluene may have entered the radioactive waste stream. The volume of this material in the waste is believed to be small. A second aspect of the project evaluated various radioisotope tags in aqueous and organic fluids, and in slurries typical of those encountered in the mining, chemical, and metallurgical industries. The evaluations were conducted in a pipeline loop that was constructed on a pilot plant level for dynamic testing of the solutions and slurries. A 6" X 6" pipe sleeve insert was neutron activated (Fe^{59}) and placed in the pipeline loop. As a particular type slurry was pumped through the pipeline, it was monitored for Fe^{59} . The values derived from the experiments were used to calculate pipeline abrasion rates for a particular type of slurry. Waste from this project generally consisted of irradiated pipe inserts, paper wipes, and broken glassware. The slurries used in the experiments were disposed of on-site at the CSMRI. It is believed no chemical waste from this aspect of the project entered the radioactive waste sent to the INEL.

During 1963 and 1964 the CSMRI conducted classified research (CSMRI Project 330412) for the Defense Atomic Support Agency (DASA), Tonopah, Nevada. The research was in support of DASA Project 2.6D, Operation ROLLER COASTER, under contract DA-49-146-XZ-225. The purpose of the project was to determine the distribution of special nuclear materials in various soil size fractions if a high explosive detonation occurs. This included possible reactions between special nuclear materials and the mineralogical constituents of the various size fractions. The CSMRI conducted petrologic and mineralogic examinations of the pre-shot soil samples. Size analysis and alpha counting procedures were conducted on post-shot soil samples.

Documents from the CSMRI indicate the plutonium contaminated wastes from the project was packaged in four (4) 55 gallon steel drums. This waste was then delivered to the Rocky Flats Plant for disposal. It can be assumed this waste was then sent to the INEL. Dry waste, such as unused portions of 25 post-shot soil samples, contaminated paper, and glassware, was placed in two (2) drums. According to F. L. Smith, there is a good possibility an unknown number of Vycor beakers were included in the waste. These beakers were used to prepare the soil samples for alpha counting procedures.

J. D. McKinney
TLC-2-80
January 22, 1980
Page 3

Each beaker may contain up to 350 ml of 4M HCl acid. The other two (2) drums contained wet wastes from solutions used to clean soil sizing screens and infrasizer cones. The solutions contained water, small amounts of Alconox (a detergent), and acetone. The solutions in both drums were treated with an unknown flocculant. The total plutonium content of all waste drums was 2.6 micrograms.

The project engineer for all of the above projects is deceased. The location of other individuals, mainly technicians, involved in the projects is unknown. Radioactive Waste disposal services were provided by the Nuclear Engineering Company, Inc., after October, 1962. Deposition of this waste occurred at Beatty, Nevada.

fg

cc: H. M. Batchelder
J. L. Clark
J. R. Fielding
K. B. McKinley
R. B. O'Brien *RLS*
R. L. Silverthorne /r/ File
Central File
T. L. Clements File (2)

Colorado School of Mines Research Institute

P.O. BOX 112 • GOLDEN, COLORADO 80401

PHONE (303) 279-2581

CSMRI

December 13, 1979

Mr. Tom Clements, Jr.
United States Department of Energy
Idaho Operations Office
550 Second Street
Idaho Falls, Idaho 83401

Dear Sir:

We are returning your waste questionnaire, which has been filled out to the best of our ability.

As I mentioned to you on the phone last week, there are some files, relative to the production of this waste, here at the Institute which you are welcome to look through if you decide on a personal visit. I could find nothing in them other than what is covered in your questionnaire.

Mr. Fred L. Smith, 8795 Ralston Road, Arvada, Colorado, 80002 was responsible for these shipments and it is possible that he could tell you more about them.

If I can be of further help with this matter please let me know.

Sincerely,

Jack E. Coulson

Jack E. Coulson, Manager
Technical Services and Construction Division

/mjm

Enc.

Colorado School of Mines Research Institute

P.O. BOX 112 • GOLDEN, COLORADO 80401
PHONE (303) 279-2581

CSMRI

January 9, 1980

RECEIVED

J. L. Clark

JAN 15 1980

Mr. J. L. Clark, Manager
Safety Standards Branch
EG&G Idaho, Inc.
P. O. Box 1625
Idaho Falls, Idaho 83415

Action of
Action
Reply to
[Handwritten signature: JLS/trc]

Dear Mr. Clark:

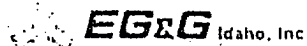
I will expect to see Mr. Clements in my office on January 15 - 16, 1980. He will have access to all available materials relative to the shipments of radioactive wastes from our firm to the Idaho National Engineering Laboratory.

Sincerely,

[Handwritten signature: Jack E. Coulson]

Jack E. Coulson, Manager
Technical Services and Construction Division

/mjm



P.O. BOX 1625, IDAHO FALLS, IDAHO 83415

January 18, 1980

Mr. Jack Coulson, Manager
Technical Services and Construction Division
Colorado School of Mines Research Institute
P O. Box 112
Golden, Colorado 80401

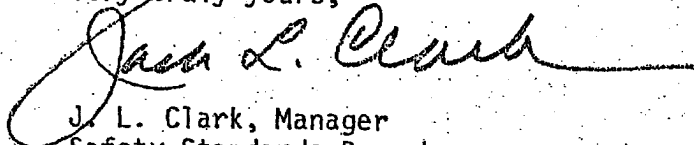
NONRADIOLOGICAL HAZARDS STUDY - JLC-21-80

Dear Mr. Coulson:

Thank you for the time and cooperation you extended to Mr. T. L. Clements on his visit to your facility on January 15-16, 1980. The waste shipment documents and project reports have been very beneficial in support of the Nonradiological Hazards Study. The help of Ms. Lola Ann Johnson in locating these documents was also appreciated.

Enclosed are copies of the waste shipment records for your file.

Very truly yours,



J. L. Clark, Manager
Safety Standards Branch

TLC:ib

Attachments:
As Stated

bcc: T. L. Clements ✓
J. R. Fielding
J. D. McKinney
R. L. Silverthorne (r) file
Central File
J. L. Clark file

PUBLIC SERVICE COMPANY

Wash. Field, Wash.

November 12, 1962

Disposal of
Off-Site Radioactive Waste

NOTECRAME

TO: Ray Fisher

FROM: A. A. Anselmo:pw

Attached herewith are Waste Shipment Data forms for the burial of radioactive waste as covered by AIB-3, 11.

SHIPPER	SERIAL NO.	CU FT BURIED	NO PIECES	DATE BURIED
Colorado School of Mines Research Foundation, Inc P. O. Box 112 Golden, Colorado	CSM-61-1	14.6	2	11-5-62

14.6 cu. ft. @ 4.70 - 4.10.22

Carrier: Garrett

minimum charge per shipment - \$21.00

Enc - as listed

cc: J. W. Latchum w/o enc
L. P. Smith w/o enc
A. A. Anselmo Etc w/enc

PURCHASE REQUISITION

REQUISITIONED BY -29-62 Kent Perry		DEPARTMENT Mining		APPROVED BY <i>[Signature]</i>	
R TO (PERSON AND DEPT.)		CHARGE TO 50% to 320311-Act. Anal.; 50% to 300301			
D FOR Radioactive waste disposal, charges for services		DATE ORDERED		PURCHASE ORDER NO. 26806	
FILLED IN BY PURCHASING DEPARTMENT ONLY		SHIP TO			
DER U.S. Atomic Energy Commission					
COM Idaho Operations Office					
c/o Phillips Petroleum Company					
P.O. Box 2067, Idaho Falls, Idaho		SHIP VIA			
B. TERMS		DELIVERY DATE		CHARGE TAX	TAX EXEMPT
QUANTITY	DESCRIPTION			PRICE	AMOUNT
	Disposal of radioactive waste material			21.00	21.00
D BY		RECEIVED BY		DATE RECEIVED	

PHILLIPS PETROLEUM COMPANY

ATOMIC ENERGY DIVISION

P.O. BOX 2067
IDAHO FALLS, IDAHO

YOUR ORDER No.
26806

Charged to:

Colorado School of Mines
Research Foundation, Inc.
P. O. Box 112
Golden, Colorado

Invoice Date 12-3-62
Invoice No. 11-104
Accounting Ref. 1470-01100
J11-1-10

Please return a copy of this
Invoice with your remittance.

DESCRIPTION

AMOUNT

For contaminated waste disposal at the NRTS burial grounds during
November, 1962 per attachment

1 shipment - minimum charge per shipment

\$21.00

C.S.M. Research Foundation
Payment Approved

Date 12-7-62
By CAW
Account 320311- 10.50
Project 300301- 10.50

POSTED
CAW

DO NOT TYPE BELOW THIS LINE

I certify that the above charges are correct and just
and payment therefore has not been received.

PHILLIPS PETROLEUM COMPANY

CHIEF ACCOUNTANT

Appendix B

Letters from Colorado School of Mines

11

UNITED STATES
ATOMIC ENERGY COMMISSION
P. O. BOX 1221
IDAHO FALLS, IDAHO

May 5, 1954

Mr. John Epp
Assistant Director, Chemical Laboratories
The Dow Chemical Company
P. O. Box 2131
Denver, Colorado

Dear Mr. Epp:

In response to your letter of April 28, requesting information relative to the condition of your van-load of contaminated waste, we are able to present the following information and observations which may be of mutual benefit in future movements of this type.

1. Truck arrived at NRTS at approximately 8:00 AM on 4/22/54, unloading and disposal were accomplished in one operation by 11:00 AM. Truck, personnel and equipment were surveyed and released by 12:00 noon.

2. An undetermined number of drums contained liquid, of these seven were leaking, five through the top and two through perforations in the bottom. At least three of these containers had leaked considerably in transit, to a degree where the liquid had soaked through the protective paper to the aluminum van floor. No detectable radioactive contamination accompanied these spills, time expended in surveying did not influence the cost of the operation. As a precautionary measure it may be advisable to use an absorbent paper on the floor of trucks carrying this type of material in the future. (Ref: - Several layers of white blotting paper, 24", B 160 caliber, .050 thickness, 25% cotton content). We are anxious to avoid disposal of liquids in our solid waste disposal ground, however, we do anticipate a certain residual amount of liquid in materials of the type that you are handling.

As a test shipment, this can be considered as exceptionally well handled by the Rocky Flats personnel. We feel that if leakage can be prevented and possible radioactive contamination of equipment and personnel is avoided that future shipments can be processed without mishap or unnecessary delay.

Yours very truly,



G. V. Beard
Chief, Health and Safety Branch
Idaho Operations Office

Appendix C

Packaging Certification by Atomic Energy Commission—ALO Contractor (August 21, 1967)

CERTIFICATION OF APPROVAL FOR FISSILE-LARGE QUANTITY SHIPPING CONTAINERS

ALBUQUERQUE OPERATIONS OFFICE, USAEC

August 21, 1967

I. ALO Contractor.

The Dow Chemical Company
Rocky Flats Division
Box 888
Golden, Colorado 80401
Contact: Traffic - W. F. Romine
Engr. - F. E. Adcock

II. Identification of Shipping Container.

Truck or trailer-on-flatcar Fissile
Class III Shipments of Radioactive Waste
in 55-gallon drums. No individual
package identification. E/E Permits
2057 and 2058 previously assigned.
ALO designation: AL-R

III. General Information Concerning Container.

Three categories of waste have been approved for shipment:

- A. Container: Used 55-gal. 18-gauge steel drums with 8-mil plastic liner.
Contents: Paper, clothing, tools and other contaminated waste generated within the Pu fabrication area but external to the dry box system.
Pu Content: 1 gram maximum, .02 grams average.
Gross Weight: 480 pounds maximum.
- B. Container: ICC-6C or -17C (or equivalent) 55-gal. steel drum with 8-mil plastic liner.
Contents: Tools, carbon molds, and other laboratory and production equipment from within the Pu dry box system (line-generated waste).
Pu Content: 200 grams maximum, 30 grams average.
Gross Weight: 880 pounds maximum.
- C. Container: ICC-17C 55-gal. drum with 8-mil plastic liner.
Contents: Hardened or semi-hardened sludges, greases, neutralized acids, and other process waste.
Pu Content: 1 to 50 grams maximum, <1 to 6 grams average, depending on the type of residue.
Gross Weight: 880 pounds maximum.

IV. Specific Limitations and Restrictions.

- 1. Sole use of vehicle required.
- 2. Average Pu content per package must not exceed 15 grams for any vehicle.
- 3. Loading to be controlled by shipper's written procedure.

V. Additional Information and/or Limitations.

None.

VI. Certification of Approval.

Pursuant to Chapter AEC 0529, this container is approved subject to the limitations described above. This certification does not relieve the shipper of his responsibility to obtain a DOT Special Permit and to comply with the requirements of other Federal Regulations as appropriate.

DATE: _____

Certification Official
Albuquerque Operations
U. S. Atomic Energy Commission

Appendix D

List of NDA Reports Available

NDA Reports Available

Date	Report/Speech Title	Authors	ID Number Report/Speech
5/31/66	Drum Counter - Washables	O. H. Willoughby	Internal Letter
4/28/69	Measurement of Plutonium in Process Materials and Contaminated Waste	O. H. Willoughby and D. R. Cartwright	RFP-1325 Speech
7/1/71 revised 9/30/71	Standardization and Performance of the Building 707 Drum Counter	R. N. Chanda, R. A. Harlan, R. A. Deal, J. L. Lawless and Y. Ferris	CRDL-950442-101 Internal Report
11/4/71	Building 771 Interim Drum Counter	R. A. Deal, R. N. Chanda, R. J. Nau R. A. Harlan and G. J. Cunningham	CRDL-950442-103 Internal Report
9/8/72	Operations Manual for Drum Counter (South) 771-A	R. A. Deal, L. A. Bidwell, J. L. Lawless, R. N. Chanda and H. R. Martin	CRDL-950442-109 Internal Report
4/26/73	Computer Control of Three Passive Assay Systems: Helix Counter, Can Counter II and South Drum Counter	J. L. Lawless and L. A. Bidwell	CRDL-950442-114 Internal Report
6/24/77	A Crate Counter for Normal Operating Loss	R. A. Harlan	RFP-2642 Speech
12/2/65	Drum Counter Verification Studies (Uranium in the Drum Counter)	O. H. Willoughby and L. D. Delpierre	CRDL-940232-101B Internal Report
12/30/65	Drum Counter Verification Studies (Plutonium/Graphite in the Drum)	O. H. Willoughby and L. D. Delpierre	CRDL-940232-101C Internal Report
5/27/66	Drum Counter Verification Studies (Plutonium/Graphite in the Drum Counter)	O. H. Willoughby and G. H. Cunningham	CRDL-940232-101D Internal Report
11/15/66	Drum Counter Evaluation Studies (Dead Time Counting Error Correction)	O. H. Willoughby and J. L. Lawless	CRDL-940232-101E Internal Report
3/15/67	Drum Counter Evaluation Studies (Discard Waste Evaluation Summary)	O. H. Willoughby	CRDL-940232-101F Internal Report
2/14/68	Drum Counter Status Report	O. H. Willoughby, J. L. Lawless and J. L. Martinez	CRDL-940232-101G Internal Report
2/16/68	Americium in the Can and Drum Counters	J. L. Lawless and O. H. Willoughby	CRDL-940232-101H Internal Report
2/27/68	Drum and Can Counter Computer Program	J. L. Lawless and O. H. Willoughby	CRDL-940232-101I Internal Report

Appendix E

Handbook of the Rocky Flats Plant Production Non-Destructive Assay Systems (compiled by Bill Ulbricht)

**HANDBOOK
OF THE
ROCKY FLATS PLANT
PRODUCTION
NON-DESTRUCTIVE
ASSAY SYSTEMS**



Rockwell International

**COMPILED BY:
BILL ULBRICHT
PCCO-NDA**

ROCKY FLATS PLANT
ENERGY SYSTEMS GROUP
P. O. Box 464
Golden, Colorado 80401
(303) 497-7000
Contractor to
U. S. Department of Energy



Rockwell
International

June 1984

Dear Reader:

This is the first attempt at this handbook. It represents an attempt to combine information about the Production NDA Counting Systems at Rocky Flats Plant into one handbook. If you notice errors, wish additions, or have other constructive comments please direct them to Bill Ulbricht, (x7644) PCCO, Bldg. 771. This is to be an on-going effort.

Bill Ulbricht

Cordially,
Bill Ulbricht
PCCO-NDA

COMMENTS:

SIGNED: _____

June 1984

NEW NAME: 707DS OLD NAME: Segmented Drum Scanner LOCATION: bldg. 707 room PHONE: 2966

PURPOSE: Pu assay of 55 gallon drums using high resolution gamma spectra. Transmission corrected.

STATUS: operational

AGE: 2 years

FUTURE:

SAMPLES: TYPE(IDC): 300, 301, 320, 328, 330, 331, 336, 337, 338, 339, 374, 376, 432, 440, 441, 442, 480, 481, 484, 485

CONTAINERS: 55 gallon drums

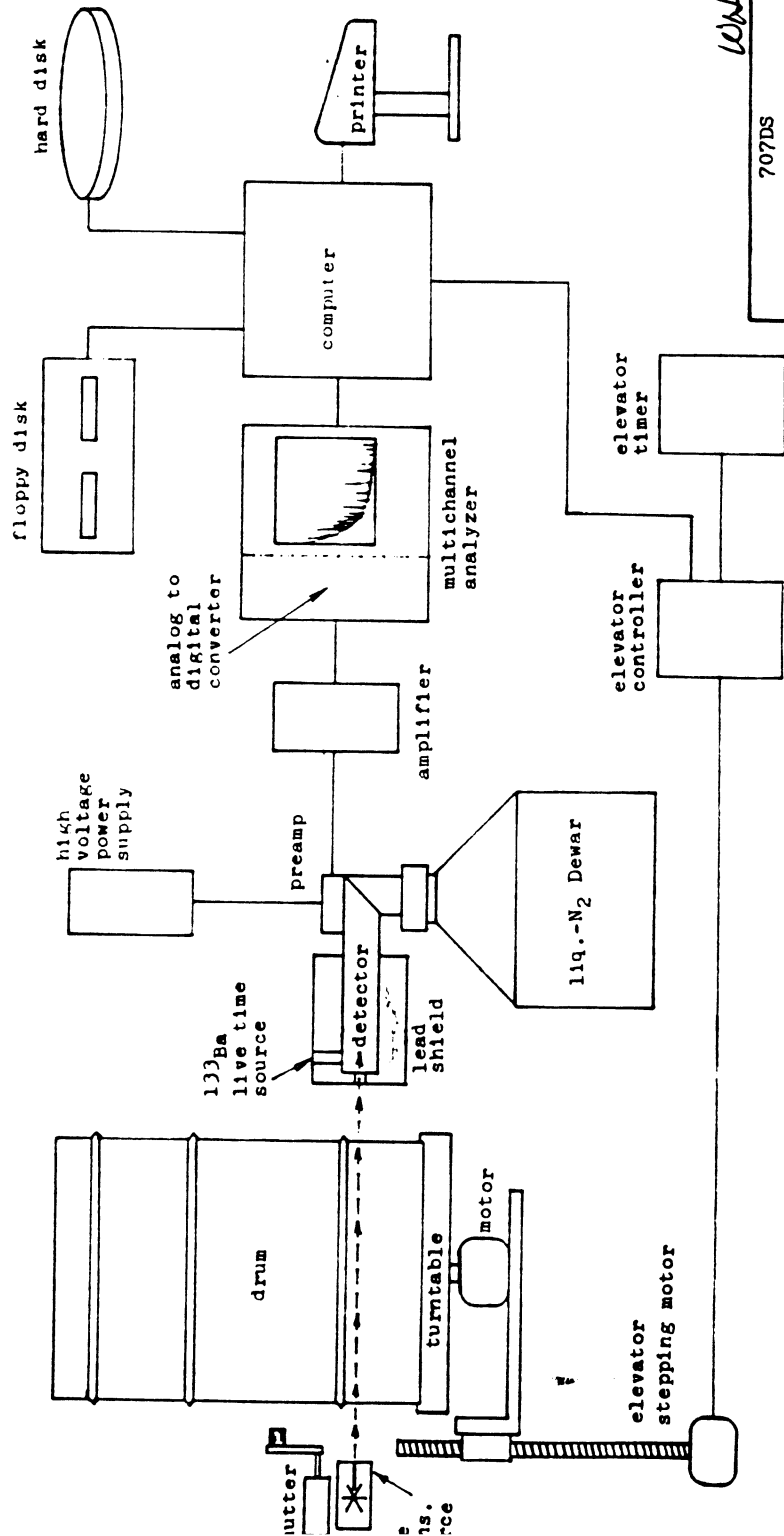
SAMPLE SIZE: 300g. MAX. 0 g. MIN. Pu
 20g. MAX. 0 g. MIN. Am

ASSAY TIME: 3 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

The elevator permits the drum to be divided into segments or slices for assay. Each segment is assayed individually for ^{239}Pu (414 keV gamma ray peak) and ^{241}Am (662 keV gamma ray peak). A transmission correction is computed for each segment based on the ^{75}Se (401 keV gamma ray peak) transmission source peak area. A total of the quantities of Pu and Am in all the segments is computed and reported at the end of the assay.



707DS

707 SEGMENTED DRUM SCANNER

WML

June 1984

NEW NAME: 771DS OLD NAME: Segmented Drum Scanner LOCATION: bldg. 771 room 301 (annex) PHONE: 2939-annex 2601-control room

PURPOSE: Pu assay of 55 gallon drums using high resolution gamma ray spectra. Transmission corrected.

STATUS: operational

AGE: 3 years

FUTURE:

SAMPLES: TYPE(IDC): 301, 302, 320, 328, 330, 331, 336, 337, 338, 339, 374, 376, 441, 442, 480, 481

CONTAINERS: 55 gallon drums

<u>SAMPLE SIZE</u> :	300 g. <u>MAX.</u>	0 g. <u>MIN.</u> Pu
	20 g. <u>MAX.</u>	0 g. <u>MIN.</u> Am
	120 g. <u>MAX.</u>	0 g. <u>MIN.</u> U

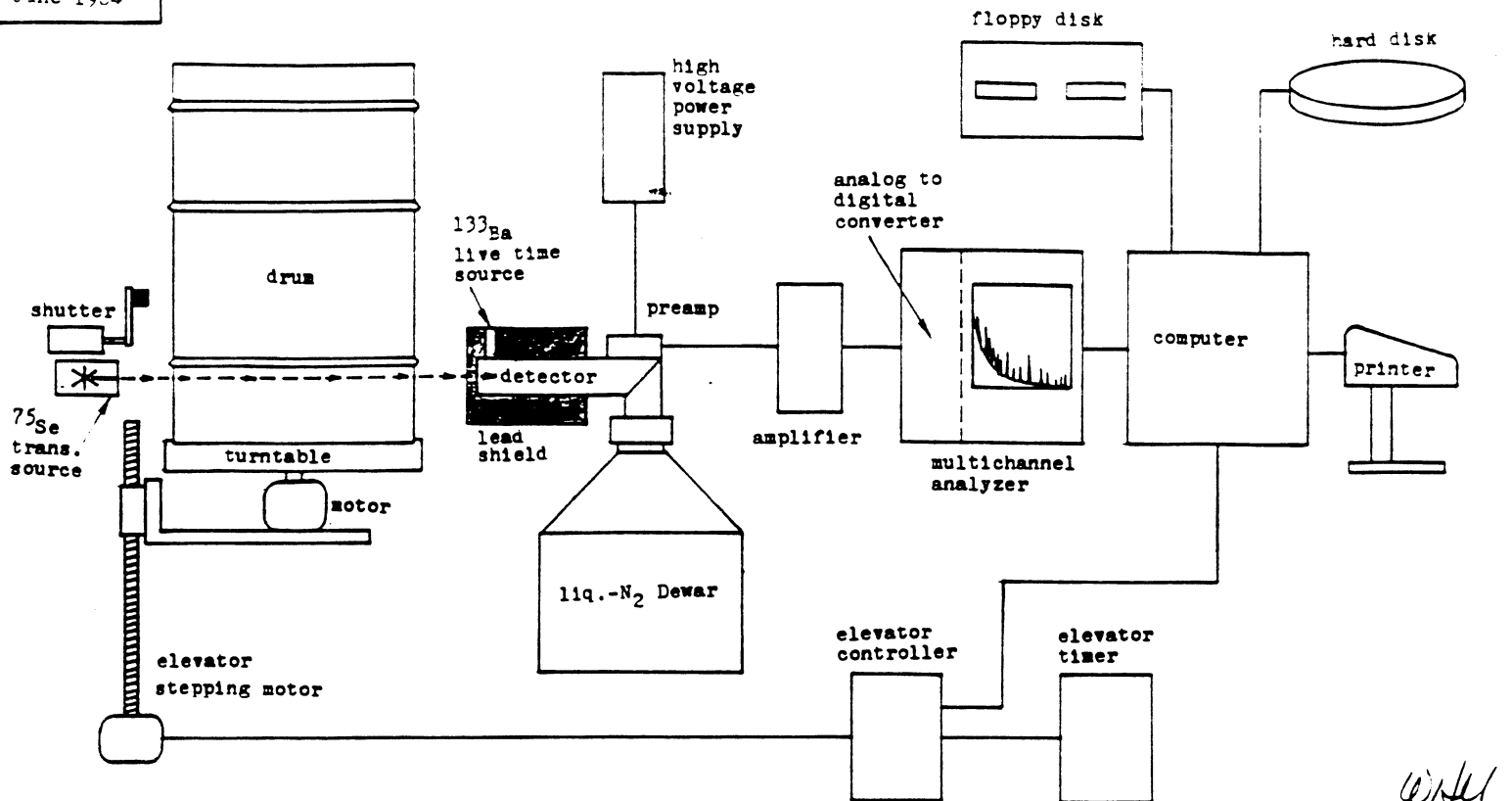
ASSAY TIME: 3 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

The elevator mechanism permits the drum to be divided into segments or slices for the gamma ray assay. Each segment is assayed individually for ^{239}Pu (414 keV) and ^{241}Am (662 keV). A transmission correction is computed for each segment based on the ^{75}Se (401 keV) transmission source peak area. A total of the quantities of Pu and Am in all the segments is computed and reported at the end of the assay.

June 1984



771DS
771 SEGMENTED DRUM SCANNER

June 1984

NEW NAME: 771CS OLD NAME: Can Scan I LOCATION: bldg. 771 room 147F PHONE: 2601

PURPOSE: Pu and Am assay of cans using high resolution gamma ray spectra. Transmission corrected.
For Molten Salts

STATUS: operational

AGE: 5 years

FUTURE:

SAMPLES: TYPE(IDC): 404, 405, 406, 407, 408, 409, 410, 411

CONTAINERS: 1 and 4 liter cans

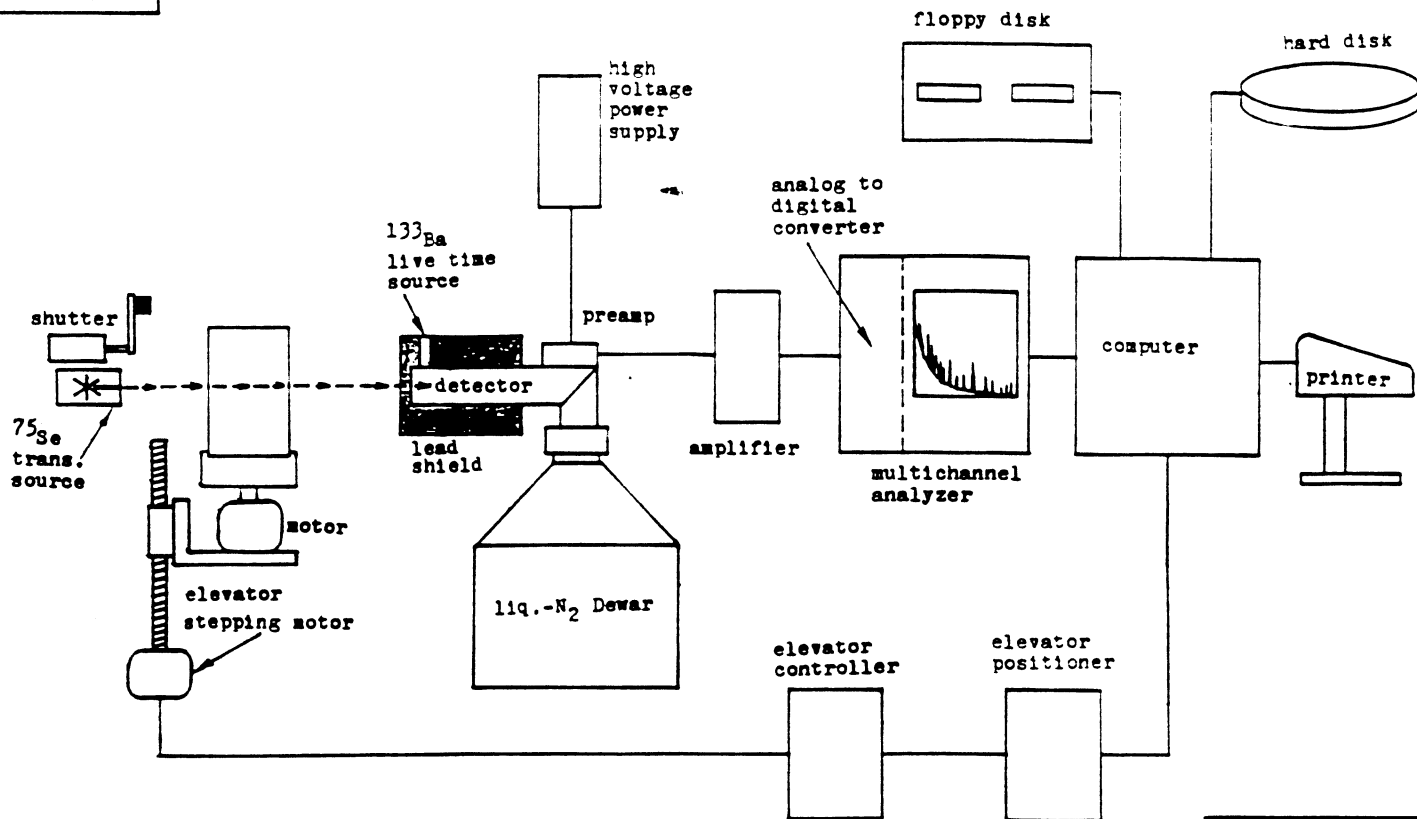
<u>SAMPLE SIZE:</u>	400 g. <u>MAX.</u>	0 g. <u>MIN.</u>	Pu
	37 g. <u>MAX.</u>	0 g. <u>MIN.</u>	Am

ASSAY TIME: 3 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

The elevator mechanism permits the can to be assayed in segments or slices. Each segment is assayed for ^{239}Pu (by examining the 414 keV peak) and ^{241}Am (662 keV). A transmission correction is computed for each segment based on the 401 keV peak (^{75}Se transmission source). A total of the quantities of Pu and Am in all the segments is computed and reported at the end of the assay.



June 1984

NEW NAME: 776CS OLD NAME: Can Scan II LOCATION: bldg. 776 room

PHONE: 2076-process
ares
4151-control
room

PURPOSE: Pu and Am assay of Molten Salts either in line or out of line by high resolution gamma ray spectroscopy. Transmission corrected.
STATUS: operational

AGE: 2 years

FUTURE:

SAMPLES: TYPE(IDC): 404, 405, 406, 407, 408, 409, 410, 411

CONTAINERS: 1 liter cans

SAMPLE SIZE: 400 g. MAX. 0 g. MIN. Pu
 37 g. MAX. 0 g. MIN. Am

ASSAY TIME: 3 samples/hour

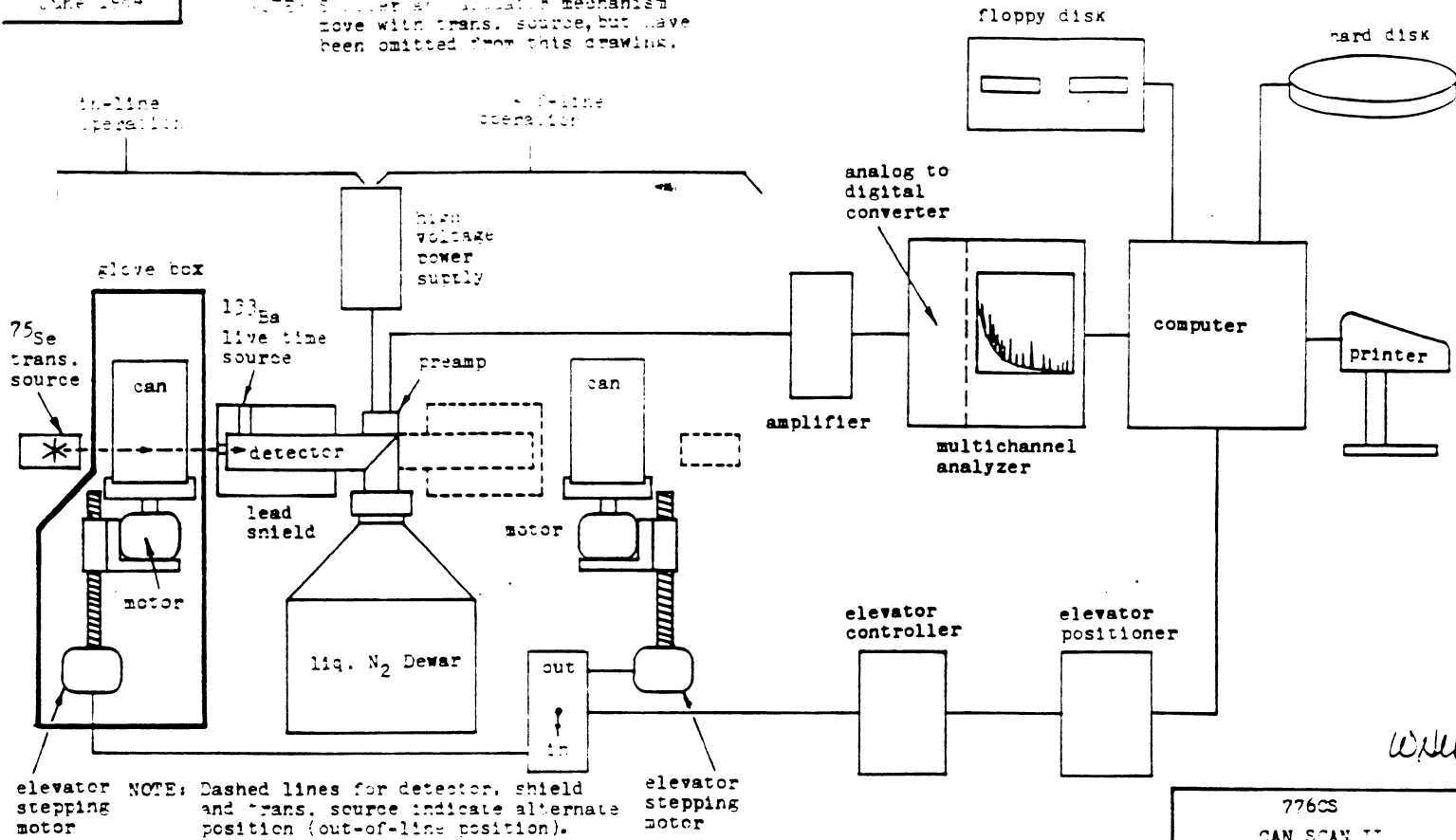
MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

The elevator mechanism permits the can to be assayed in segments or slices. Each segment is assayed for ^{239}Pu by examining the 414 keV peak and ^{241}Pu with the 662 keV peak. A transmission correction is computed for each segment based on the 401 keV peak from the ^{75}Se transmission source. A total of the quantities of Pu and Am in all the segments is computed and reported at the end of the assay.

June 1984

NOTE: Scanner and elevation mechanism move with trans. source, but have been omitted from this drawing.



June 1984

NEW NAME: 371CS1 OLD NAME: Can Scan III LOCATION: bldg. 371 room 3305(det) PHONE: 4782-control
room 3315(computer) room

PURPOSE: Pu and Am assay of Electrorefined Salts by high resolution gamma ray spectroscopy.
Transmission corrected.

STATUS: operational

AGE: 2 years

FUTURE: replace Nuclear Data data acquisition system with a DEC
computer and Canberra multichannel analyzer so that this
unit can use standardized software.

SAMPLES: TYPE(IDC): 409, 411

CONTAINERS: tall stacker/retriever cans (2.75 l.)

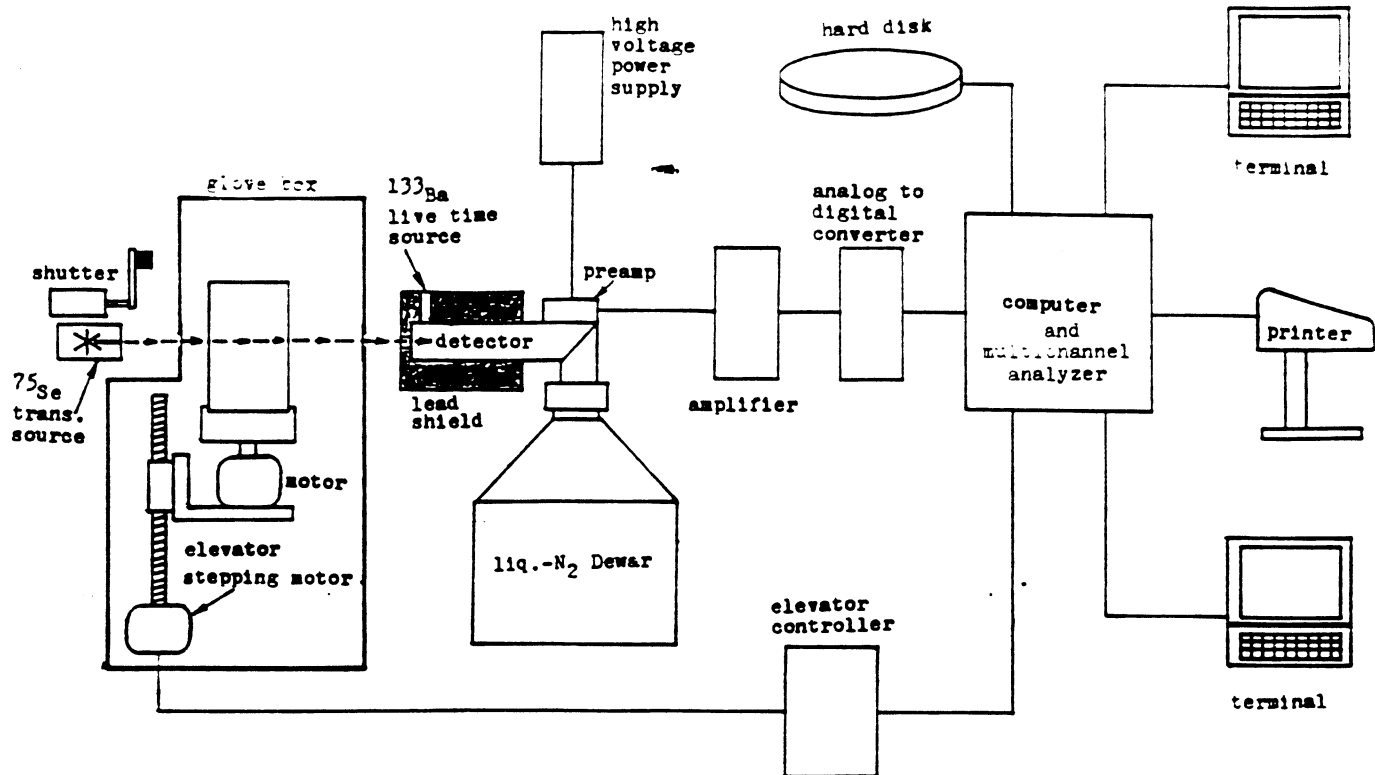
<u>SAMPLE SIZE</u> :	280 g. <u>MAX</u> .	0 g. <u>MIN</u> . Pu
	5 g. <u>MAX</u> .	0 g. <u>MIN</u> . Am

ASSAY TIME: 3 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

The elevator mechanism permits the can to be assayed in segments or slices. Each segment is assayed for ^{239}Pu and ^{241}Am by examining the 414 keV and 662 keV peaks respectively. A transmission correction is computed for each segment based on the area of the 401 keV peak from the ^{75}Se transmission source. A total of the quantities of Pu and Am in all the segments is computed and reported at the end of the assay.



June 1984

NEW NAME: 371CS2 OLD NAME: Can Scan IV LOCATION: bldg. 371 room 3341 PHONE:

PURPOSE: Pu assay of residues for offsite shipment by high resolution gamma ray spectroscopy.
Transmission corrected.

STATUS: operational

AGE: 3 months

FUTURE:

SAMPLES: TYPE(IDC): 392, 409, 420

CONTAINERS: 1 liter

<u>SAMPLE SIZE</u> :	400 g. <u>MAX</u> .	0 g. <u>MIN</u> . Pu
	37 g. <u>MAX</u> .	0 g. <u>MIN</u> . Am

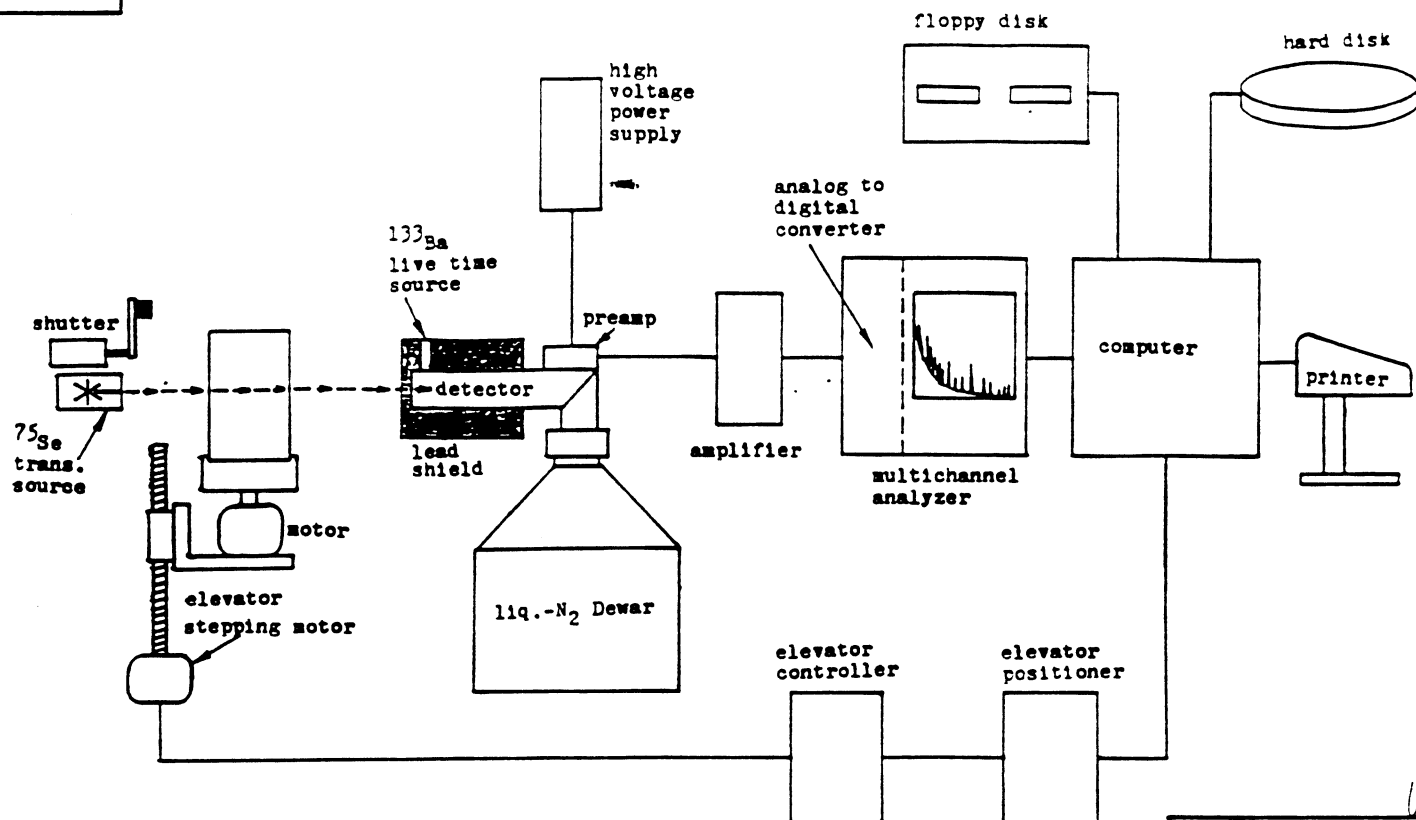
ASSAY TIME: 3 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

The elevator mechanism permits the can to be assayed in segments or slices. Each segment is assayed for ^{239}Pu and ^{241}Am by examining the 414 keV and 662 keV peaks respectively. A transmission correction is computed for each segment based on the area of the 401 keV peak from the ^{75}Se transmission source measured for each segment. A total of the quantities of Pu and Am in all the segments is computed and reported at the end of the assay.

June 1984



371CS2
CAN SCAN IV

June 1984

NEW NAME: 371DS OLD NAME: Segmented Drum Scanner LOCATION: bldg. 371 room PHONE:

PURPOSE: Pu assay of 55 gallon drums using high resolution gamma ray spectra. Transmission corrected.

STATUS: to be ordered

AGE: _____ FUTURE: _____

SAMPLES: TYPE(IDC):

CONTAINERS: 55 gallon drums

SAMPLE SIZE: g. MAX. g. MIN.

ASSAY TIME: 3 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

The elevator mechanism permits the drum to be assayed in segments or slices. Each segment is assayed for ^{239}Pu and ^{241}Am by examining the 414 keV and 662 keV peaks respectively. A transmission correction is computed for each segment based on the area of the 401 keV peak from the ^{75}Se transmission source measured for each segment. A total of the quantities of Pu and Am in all the segments is computed and reported at the end of the assay.

**LOW RESOLUTION
GAMMA SYSTEMS**

June 1984

NEW NAME: 771CA1 OLD NAME: Can Counter LOCATION: bldg. 771 room 147A PHONE: 2601

PURPOSE: Pu assay by low resolution gamma ray detector (sodium iodide)

STATUS: operational

AGE: 14 years

FUTURE: to be replaced with can scan system eventually

SAMPLES: TYPE(IDC): 062, 290, 310, 311, 312, 320, 328, 330, 331, 332, 333, 334, 336, 337, 338,
339, 340, 369, 370, 371, 372, 373, 375, 376, 377, 378, 390, 391, 392, 393,
396, 397, 398, 411, 420, 421, 422, 423, 430, 431, 440, 441, 442, 480, 481
CONTAINERS: $\frac{1}{2}$ or 1 gal. poly bottle, 8801 or 8802 volrath can, 140 g. freezette

SAMPLE SIZE: 200 g. MAX. 0.5 g. MIN.

ASSAY TIME: 15 samples/hour

MEASUREMENT ACCURACY: \pm 20%

DESCRIPTION OF ASSAY:

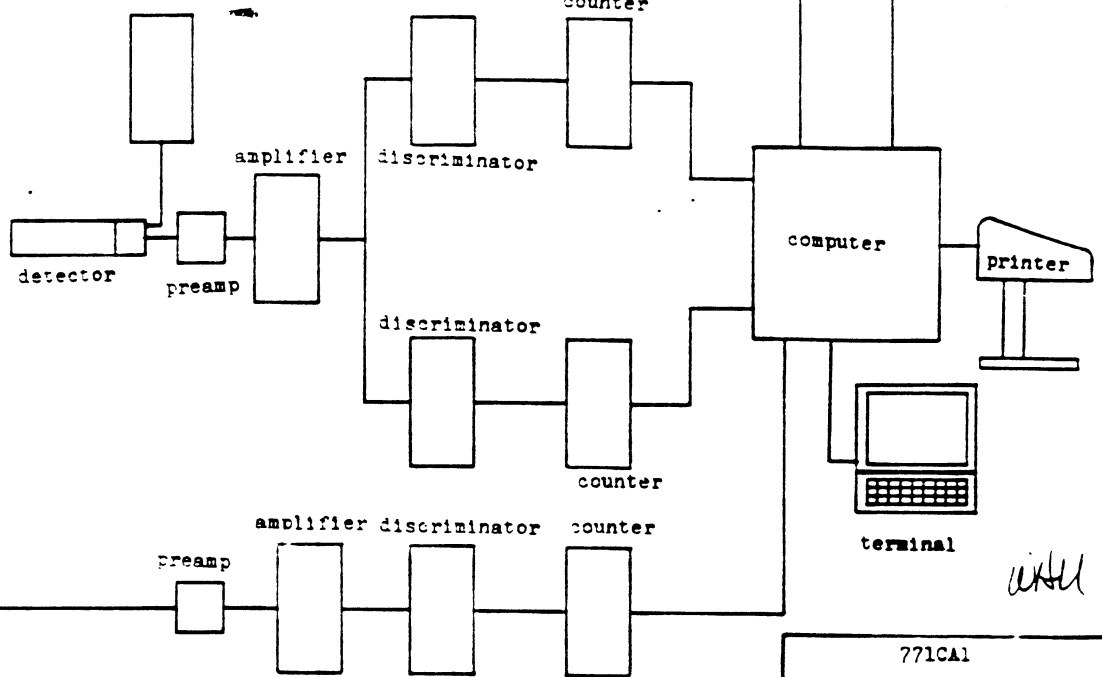
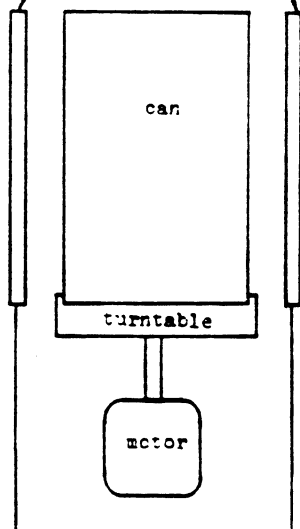
The assay consists of a 100 sec. count. Two single channel analyzers are used. One detects Compton and background activity and the other Pu activity. Background and Compton counts are computed from the two values taken above and subtracted from the Pu activity counts to yield a final count. A gamma ray attenuation factor based upon prior work and assigned to the sample's IDC is applied to the final count. This product is proportional to the amount of ^{239}Pu in the sample.

The neutron activity is measured and is considered only when the gamma value and neutron value disagree by a large factor.

June 1984

paper tape reader/punch

EP₃ neutron detectors (6 total)



771CA1
CAN COUNTER

June 1984

NEW NAME: 771CA2 OLD NAME: Helix Counter LOCATION: bldg. 771 room 147A PHONE: 2601

PURPOSE: Pu assay by low resolution gamma ray detector (sodium iodide). Transmission corrected.

STATUS: operational

AGE: 14 years

FUTURE:

SAMPLES: TYPE(IDC): 338, 396, 397, 398, 420, 421, 422, 423

CONTAINERS: $\frac{1}{2}$ gal. and 1 gal. wide mouth poly bottle in clam shell

SAMPLE SIZE: 500 g. MAX. 0 g. MIN.

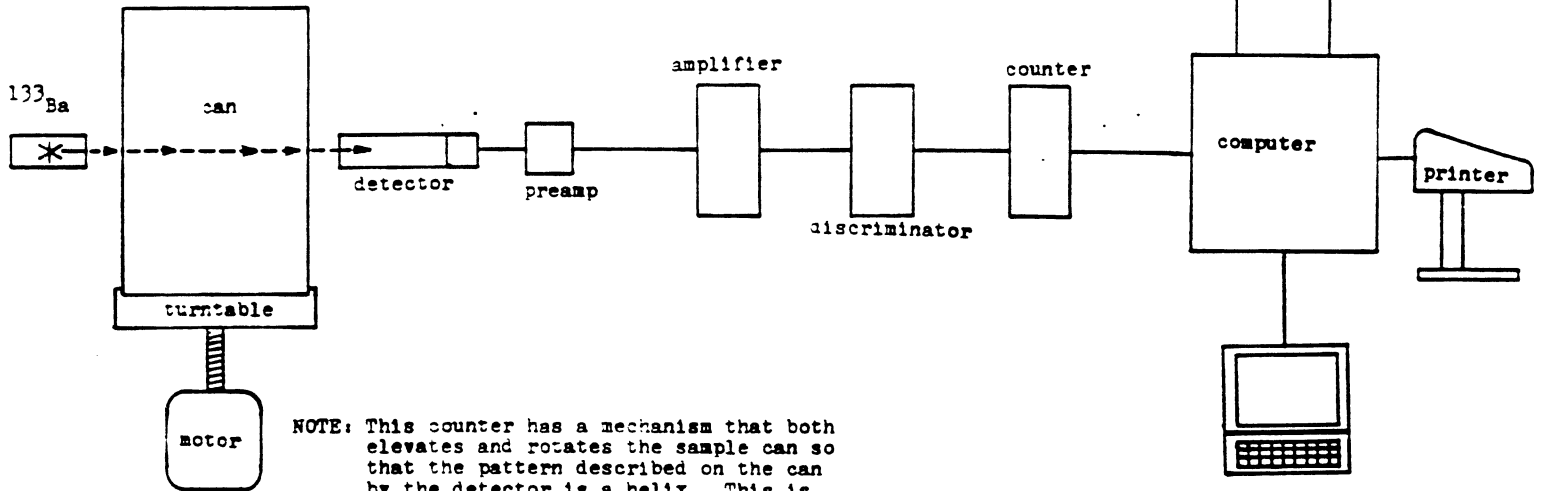
ASSAY TIME: 10 samples/hour

MEASUREMENT ACCURACY: 3% on IDC 420

DESCRIPTION OF ASSAY:

The helix counter is a low resolution counter that scans the sample in a helical pattern. It also utilizes a ^{133}Ba transmission source for the attenuation correction. Four 100 sec. counts are taken for each sample: (1) background, (2) background plus transmission source activity, (3) sample, and (4) sample plus transmission source. Only one counter is used and it takes data on the 384 complex of gamma rays. This assay is only applicable to full containers, otherwise the transmission correction factor will be in error.

133Ba



NOTE: This counter has a mechanism that both elevates and rotates the sample can so that the pattern described on the can by the detector is a helix. This is to assure that all parts of the can are seen by the detector.

terminal

771CA2
HELIX COUNTER

June 1984

NEW NAME: 771DA OLD NAME: South Drum Counter LOCATION: bldg. 771 room 304 PHONE: 2939
(annex)

PURPOSE: Pu assay by low resolution gamma ray detector (sodium iodide) for 55 gallon drums

STATUS: operational

AGE: 14 years

FUTURE: upgrade

SAMPLES: TYPE(IDC): 292, 300, 301, 302, 320, 328, 330, 331, 334, 335, 336, 337, 338, 339, 370,
371, 372, 374, 375, 376, 377, 378, 425, 430, 431, 432, 440, 441, 442, 480,
481, 490 ✓

CONTAINERS: 55 gallon drums

SAMPLE SIZE: 300 g. MAX. g. MIN.

ASSAY TIME: 25 samples/hour

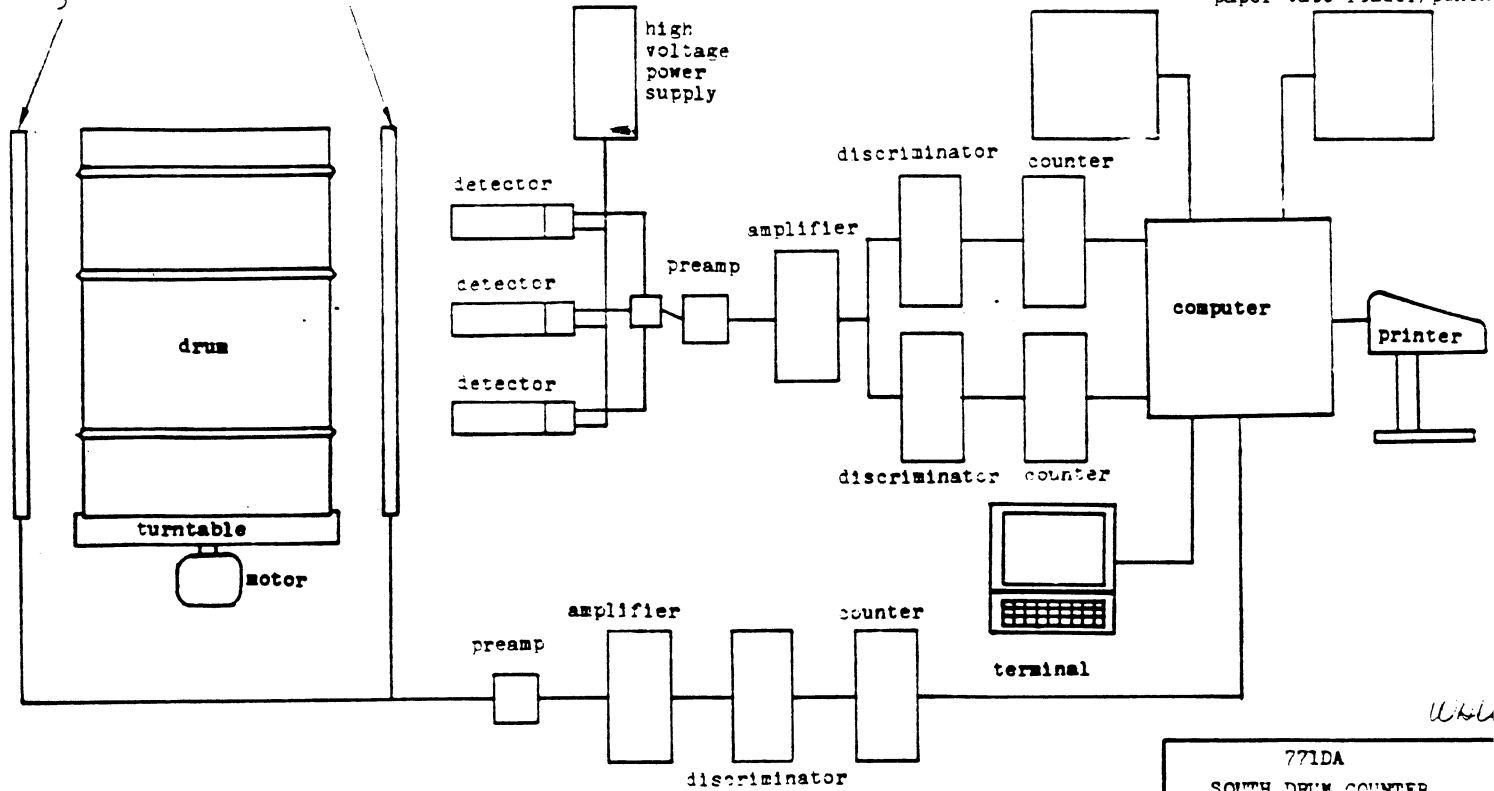
MEASUREMENT ACCURACY: 30%

DESCRIPTION OF ASSAY:

The assay consists of a 100 sec. count. Two single channel analyzers are used. One collects counts from the background and Compton regions of the spectrum while the other measures counts in the region of the 384 complex of gamma ray peaks. The Compton and background counts are subtracted from the 384 peak complex and the the peak area is computed. A gamma ray attenuation factor based upon prior determinations of attenuation factors for samples of the same IDC is applied with the 384 complex peak area to compute the Pu in the sample.

The neutron activity is measured and is considered only when the gamma value for the Pu in the sample and the neutron value for the Pu disagree by a large factor.

BF₃ neutron detectors (12 total)



771DA
SOUTH DRUM COUNTER

June 1984

NEW NAME: 776DA

OLD NAME: "LCSAC"

LOCATION: bldg. 776

PHONE:

PURPOSE: Pu assay to 100 nCi/g. of sample (alpha activity) using low resolution gamma ray method

STATUS: built but not yet installed or tested

AGE: 0 years

FUTURE:

SAMPLES: TYPE(IDC):

CONTAINERS: 55 gallon drums

SAMPLE SIZE:

g. MAX.

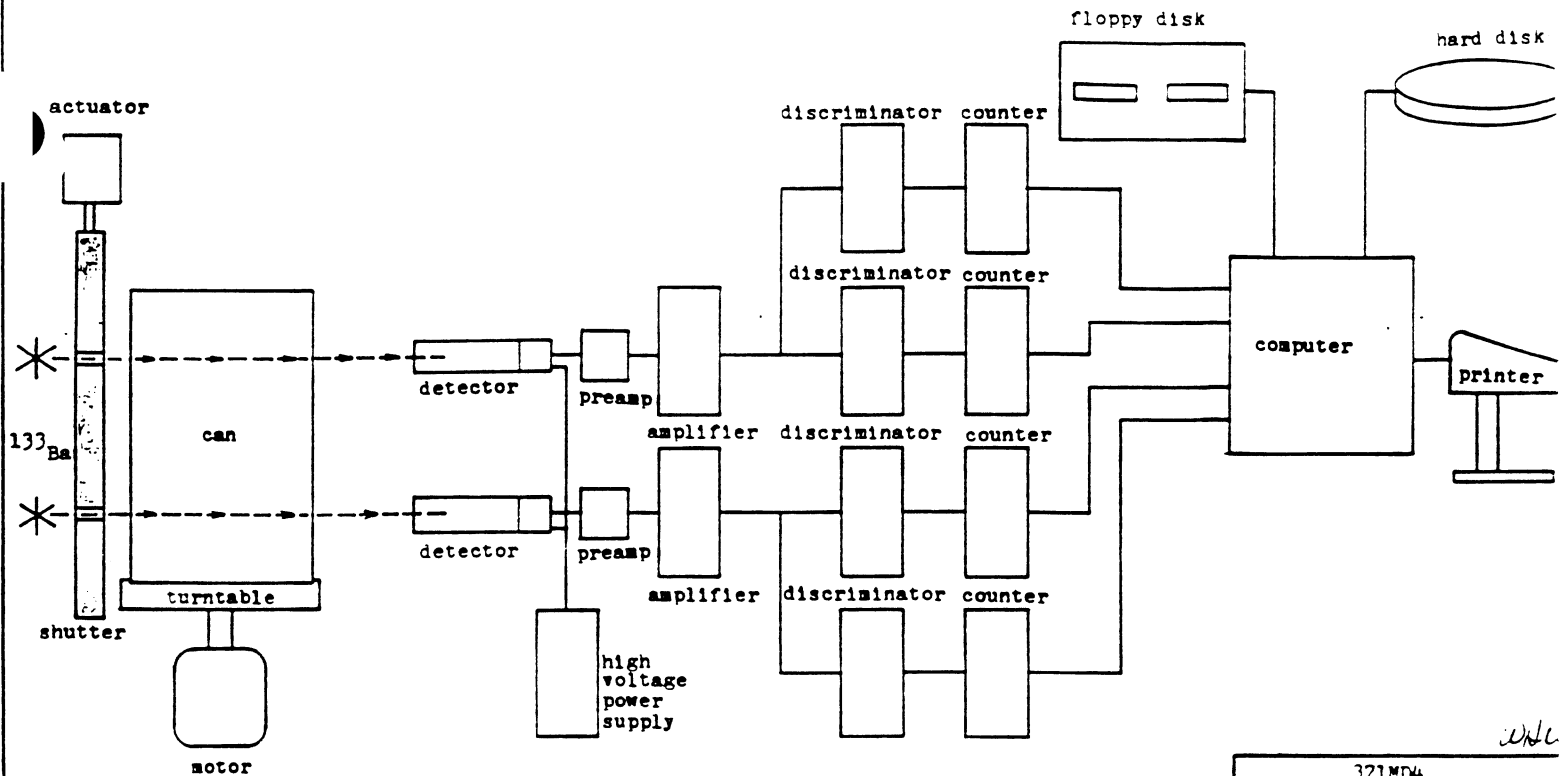
g. MIN.

ASSAY TIME: 3-5 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

Segmented assay of drums using large area sodium iodide detector with transmission source for matrix corrections.



371MD4
ML-28

June 1984

NEW NAME: 371CA4 OLD NAME: MD-28 LOCATION: bldg. 371 room PHONE:

PURPOSE: In line Pu assay using low resolution gamma ray detector (sodium iodide). Transmission corrected.

STATUS: operational

AGE: 9 years

FUTURE: replace with a segmented gamma scanner

SAMPLES: TYPE(ISO): 062, 290, 299, 300, 310, 312, 320, 330, 331, 332, 333, 336, 337, 338, 339, 340, 371, 372, 377, 378, 390, 391, 392, 393, 394, 395, 396, 397, 398, 399, 420, 421, 422, 423, 425, 431, 440, 441, 442, 480, 481

CONTAINERS: tall stacker/retriever cans (2.75 l.)

SAMPLE SIZE: 200 g. MAX. 0 g. MIN.

ASSAY TIME: 7 samples/hour

MEASUREMENT ACCURACY: $\pm 15\%$

DESCRIPTION OF ASSAY:

This counter has two low resolution detectors (NaI) and a transmission source for matrix attenuation corrections for each detector. Both detectors have two discriminators each. One discriminator for each detector is set for Compton and background and the other is set for the 384 gamma ray complex of ^{239}Pu . From these values a 384 complex peak value is computed without the Compton and background error. A peak value is reported for both detectors.

Four 60 sec. counts are taken for each assay: (1) background, (2) background plus transmission source, (3) sample only, and (4) sample plus transmission source.

This assay is applicable only to full containers, otherwise the transmission correction will be in error.

June 1984

NEW NAME: 371CA3 OLD NAME: MD-22 LOCATION: bldg. 371 room PHONE:

PURPOSE: In line Pu assay using low resolution gamma ray detector (sodium iodide). Transmission corrected.

STATUS: not operational

AGE: 9 years

FUTURE: replace with segmented gamma scanner

SAMPLES: TYPE(IDC): 062, 290, 299, 300, 310, 312, 320, 330, 331, 332, 333, 336, 337, 338, 339, 340, 371, 372, 377, 378, 390, 391, 392, 393, 394, 395, 396, 397, 398, 399, 420, 421, 422, 423, 425, 431, 440, 441, 442, 480, 481

CONTAINERS: tall stacker/retriever cans (2.75 l.)

SAMPLE SIZE: 200 g. MAX. 0g. MIN.

ASSAY TIME: 7 samples/hour

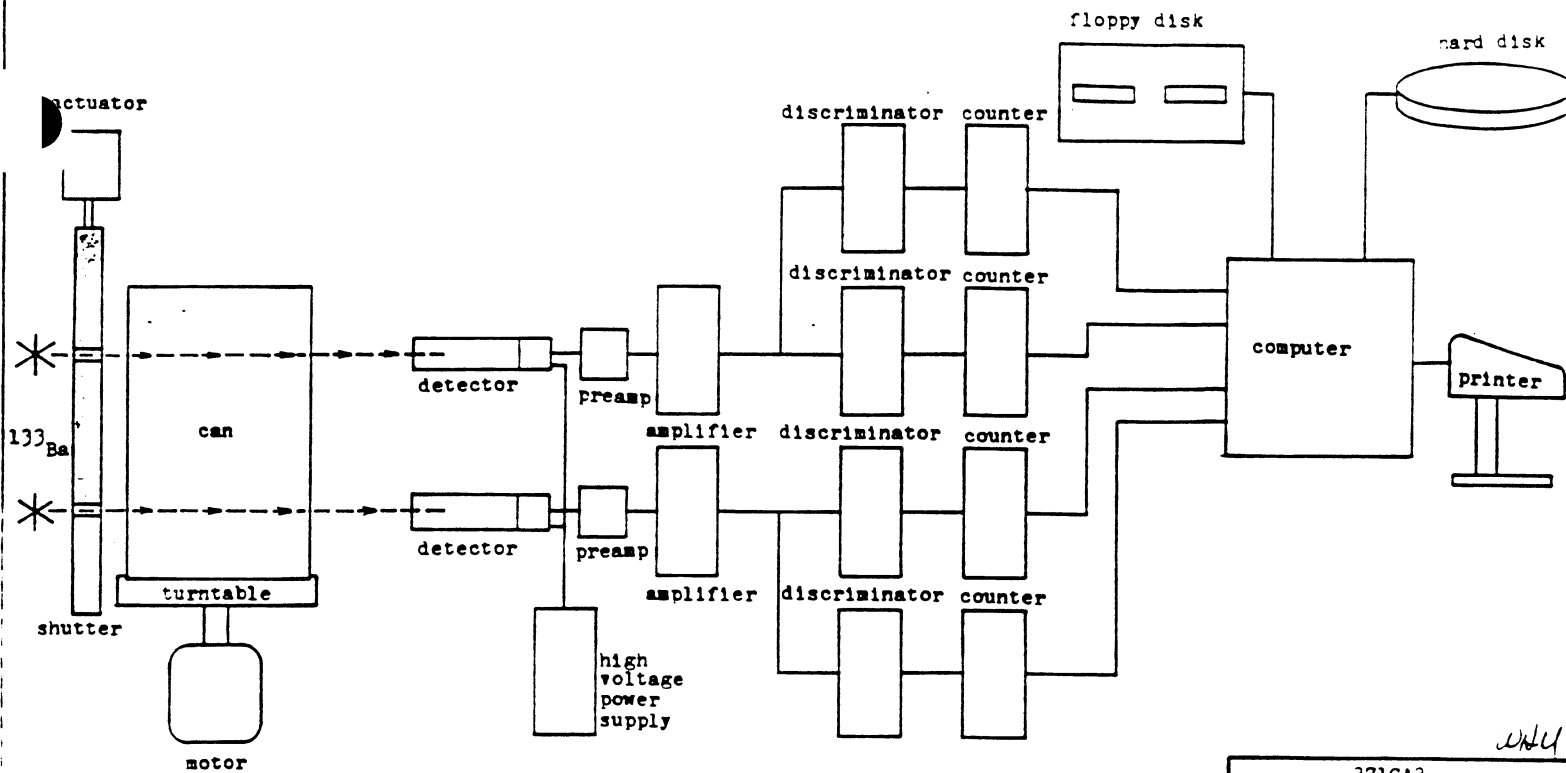
MEASUREMENT ACCURACY: $\pm 15\%$

DESCRIPTION OF ASSAY:

This counter has two low resolution detectors (NaI) and a transmission source for matrix attenuation corrections for each detector. Both detectors have two discriminators each. One discriminator for each detector is set for Compton and background and the other is set for the 384 gamma ray complex of ^{239}Pu . From these values a 384 complex peak value is computed without the Compton and background error. A peak value is reported for both detectors.

Four 60 sec. counts are taken for each assay: (1) background, (2) background plus transmission source, (3) sample only, and (4) sample plus transmission source.

This assay is applicable only to full containers, otherwise the transmission correction will be in error.



371CA3
MD-22

June 1984

NEW NAME: 371CA2 OLD NAME: MD-21

LOCATION: bldg 371 room

PHONE:

PURPOSE: In line Pu assay using low resolution gamma ray detector (sodium iodide). Transmission
STATUS: not operational corrected.

AGE: 9 years

FUTURE: to be replaced with segmented can scanner

SAMPLES: TYPE(IDC):

CONTAINERS: tall stacker retriever cans (2.75 l.)

SAMPLE SIZE: g. MAX. g. MIN.

ASSAY TIME: 7 samples/hour

MEASUREMENT ACCURACY: $\pm 15\%$

DESCRIPTION OF ASSAY:

This counter has two low resolution detectors (NaI) and a transmission source for matrix attenuation corrections for each detector. Both detectors have two single channel analyzers (discriminators) each. One discriminator for each detector is set for Compton and background and the other is set for the ^{384}Pu gamma ray complex of ^{239}Pu . From these values a 384 complex peak value is computed without the Compton and background error. A peak value is reported for both detectors.

Four 60 sec. counts are taken for each assay: (1) background, (2) background plus transmission source, (3) sample only, and (4) sample plus transmission source.

This assay is applicable only to full containers, otherwise the transmission correction will be in error.

June 1984

NEW NAME: 371CA1 OLD NAME: MD-16

OLD NAME: MD-16

LOCATION: bldg. 371 room 3515
control room 3513

PHONE: 4077-control
room

PURPOSE: In line Pu assay using low resolution gamma ray detector (sodium iodide). Transmission corrected.

STATUS: operational

AGE: 9 years

FUTURE: to be replaced with segmented can scanner

SAMPLES: TYPE (IDC): 062, 290, 299, 300, 310, 312, 320, 330, 331, 332, 333, 336, 337, 338, 339,
 340, 371, 372, 377, 378, 390, 391, 392, 393, 394, 395, 396, 397, 398, 399,
 420, 421, 422, 423, 425, 431, 440, 441, 442, 480, 481
CONTAINERS: tall stacker/retriever can (2.75 l.)

SAMPLE SIZE: 200 g. MAX. 0 g. MIN.

ASSAY TIME: 7 samples/hour

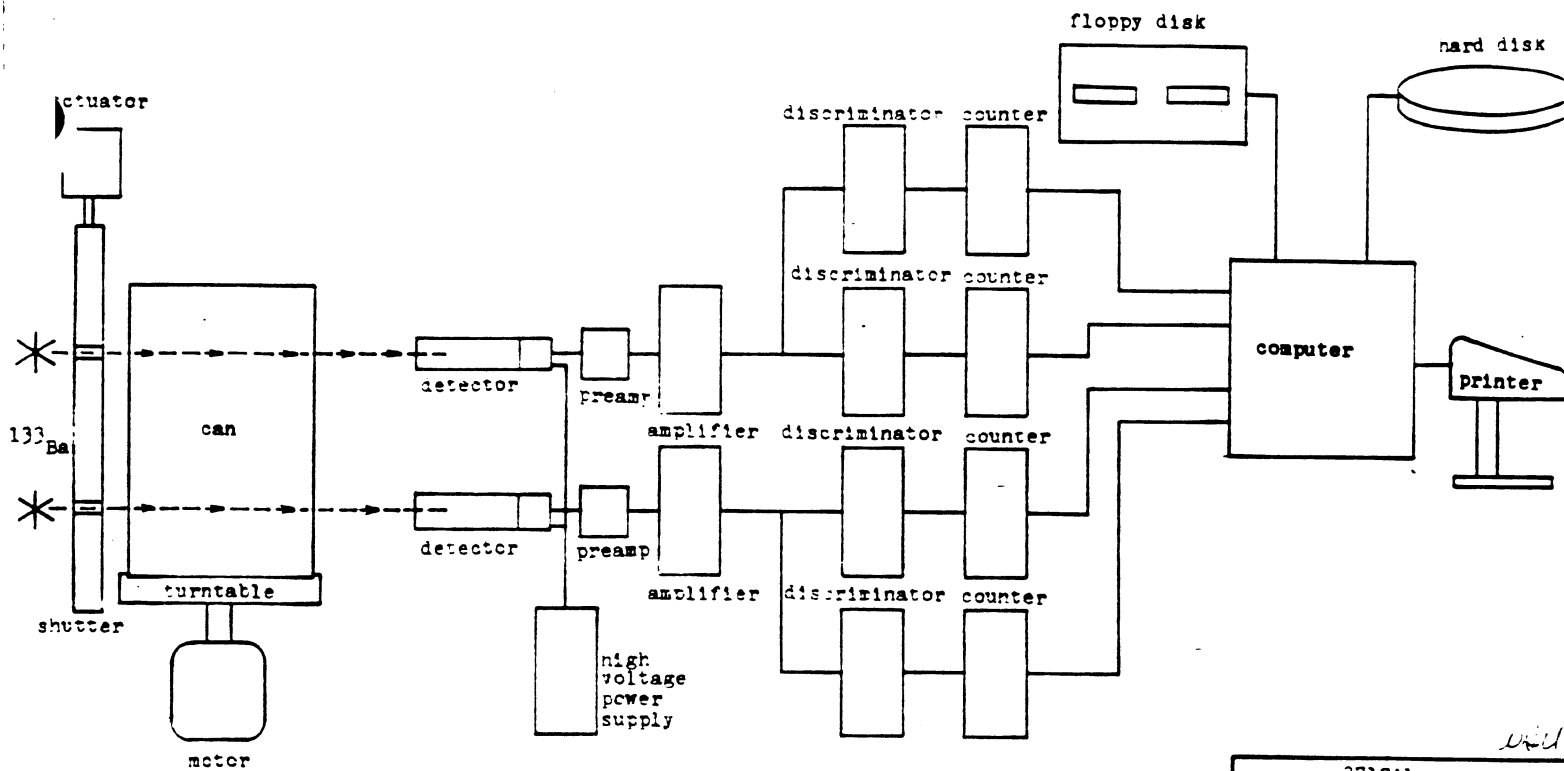
MEASUREMENT ACCURACY: $\pm 15\%$

DESCRIPTION OF ASSAY:

This counter is for residues in the button breakout area of bldg. 371. It has two low resolution detectors (NaI) and a transmission source for matrix attenuation for each detector. Both detectors have two single channel analyzers (discriminators) each. One discriminator for each detector is set for Compton and background and the other discriminator for each detector is set for the 384 gamma ray peak complex. From these values a 384 peak complex value is computed without the Compton and background interferent. A peak value is reported for both detectors. Four 60 sec. counts are taken for each assay: (1) background, (2) background plus transmission source, (3) sample only, and (4) sample plus transmission source.

This assay is applicable only to full containers, otherwise the transmission correction for the top detector will be in error.

June 1984



371CA1
MD-16

June 1984

NEW NAME: 664CrN1 OLD NAME: Old Crate Counter LOCATION: bldg. 664

PHONE: 2411-office
7368-warehouse
7643-control
room

PURPOSE: accountability by passive neutron and gamma ray assay

STATUS: operational

AGE: 5 years

FUTURE:

SAMPLES: TYPE(IDC): 330, 337, 480, 481, 490

CONTAINERS: 4' x 4' x 7' metal or wooden crates

SAMPLE SIZE: 350 g. MAX. 2 g. MIN.

ASSAY TIME: 1 samples/hour

MEASUREMENT ACCURACY: error to within a factor of 2

DESCRIPTION OF ASSAY:

Coincident neutrons from the spontaneous fissioning of ^{240}Pu are detected. The number of coincident neutrons is proportional to the amount of ^{240}Pu present.

The gamma ray assay is an independent system for determining the total amount of Pu in the crates.

Both the neutron assay and the gamma ray assay suffer from a variable background because of the location.

June 1984

NEW NAME: 664CrN2 OLD NAME: New Crate Counter LOCATION: bldg. 664
PACC

PHONE: 2411-office
7368-warehouse
7643-control
room

PURPOSE: Pu assay to separate low level waste from TRU waste

STATUS: installed, still in testing phase, not yet operational

AGE: 1 year

FUTURE:

SAMPLES: TYPE(IDC): 330, 337, 480, 481, 490

CONTAINERS: 4' x 4' x 7' metal or wooden crates

SAMPLE SIZE:

g. MAX.

g. MIN.

ASSAY TIME: 2 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

³He tubes (neutron detectors) are located in the walls of the assay chamber to completely surround the crate when the chamber is closed.

In the passive mode coincident neutrons from the spontaneous fissioning of ²⁴⁰Pu are detected for the analytical determination. The sensitivity for this mode is predicted to be about 10 mg of ²⁴⁰Pu.

The active portion of the detection system uses a pulsed neutron generator and detects prompt neutrons from induced fissions of ²³⁹Pu with special detectors (shielded ³He tubes).

The sensitivity of this method is predicted to be about 1 mg. ²³⁹Pu.

Both assay modes will be matrix dependent. In addition, both methods suffer from a variable neutron background because of the instrument's location.

June 1984

NEW NAME: 771DN OLD NAME: North Drum Counter LOCATION: bldg. 771 room 301 PHONE: 2939
(annex)

PURPOSE: Pu assay of drums by passive and/or active neutron methods

STATUS: being installed in bldg. 771 north drum counter well

AGE: 0 years.

FUTURE:

SAMPLES: TYPE(IDC): 320, 339, 480, 484, 485

CONTAINERS: 55 gal. drums

SAMPLE SIZE: g. MAX. g. MIN.

ASSAY TIME: 3-4 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

In the passive mode coincident neutrons from the spontaneous fissioning of ^{240}Pu are detected for the analytical determination. Sensitivity for this method is predicted to be 100 nCi/g. of sample.

The active mode uses a pulsed neutron generator and detects prompt neutrons from ^{239}Pu fissions with special detectors (shielded ^3He tubes). The sensitivity of this method is predicted to be 1 nCi/g. of sample.

Both methods will be matrix dependent.

June 1984

NEW NAME: 371DN OLD NAME: Drum Counter LOCATION: bldg 371 room PHONE:

PURPOSE: Pu assay of drums by passive and/or active neutron methods for Waste Operations

STATUS: to be ordered

AGE:

FUTURE:

SAMPLES: TYPE(IDC):

CONTAINERS: 55 gallon drums

SAMPLE SIZE: g. MAX. g. MIN.

ASSAY TIME: 3-4 samples/hour

MEASUREMENT ACCURACY:

DESCRIPTION OF ASSAY:

In the passive mode coincident neutrons from the spontaneous fissioning of ^{240}Pu are detected for the analytical determination. Sensitivities for this method are predicted to be on the order of 100 nCi/g. of sample.

The active mode uses a pulsed neutron generator and detects prompt neutrons from the induced fissioning of ^{239}Pu with special detectors (shielded ^3He tubes). The sensitivity of this method is estimated to be of the order of 1 nCi/g. of sample.

Both methods will be matrix dependent.

June 1984

NEW NAME: 707CN OLD NAME: Foundry PuO₂ → LOCATION: bldg. 707 module J PHONE:
Neutron Coincidence Counter Corridor

PURPOSE: Pu assay of PuO₂ after calcining by coincidence neutron method

STATUS: not yet operational

AGE: 1 year

FUTURE:

SAMPLES: TYPE(IDC): 060

CONTAINERS: short stacker/retriever cans (0.7 l.)

SAMPLE SIZE: 2500 g. MAX. 0 g. MIN.

ASSAY TIME: 12 samples/hour

MEASUREMENT ACCURACY: ± 2%

DESCRIPTION OF ASSAY:

Coincident neutrons from the spontaneous fissioning of ²⁴⁰Pu are detected. The number of coincident neutrons from the sample is related to the amount of ²⁴⁰Pu present. Isotopic values for plutonium are needed for a total Pu determination. Matrix effects could cause errors.

June 1984

NEW NAME: 371CN OLD NAME: MD-30
Calcined Oxide LOCATION: bldg. 371 room 3511 PHONE:
Coincidence Counter

PURPOSE: Pu assay of PuO_2 from calciner by coincident neutron method

STATUS: not calibrated, not yet operational

AGE: 3 years

FUTURE:

SAMPLES: TYPE(IDC): 067

CONTAINERS: short stacker/retriever cans (0.7 l.)

SAMPLE SIZE: 2000 g. MAX. 0 g. MIN.

ASSAY TIME: 12 samples/hour

MEASUREMENT ACCURACY: $\pm 2\%$

DESCRIPTION OF ASSAY:

Coincident neutrons from the spontaneous fissioning of ^{240}Pu are detected. The number of coincident neutrons from the sample is related to the amount of ^{240}Pu present. The isotopic ratios for plutonium is needed for a total Pu determination. Matrix effects could cause errors.

APPENDIX

JUNE 1984			ASSAY METHOD				APPROX. % UPTIME	COMMENTS
BLDG.	NEW NAME	OLD NAME	HI RES.	LO RES.	PASS. n°	ACT. n°		
(1)	371	371CS1	CAN SCAN III	*				
(1)	371	371CS2	CAN SCAN IV	*				
(1)	371	371CA1	MD-16		*			
(2)	371	371CA2	MD-21		*			
(2)	371	371CA3	MD-22		*			
(1)	371	371CA4	MD-28		*			
(2)	371	371CN	COINCIDENCE COUNTER			*		
(5)	371	371DS	SEGMENTED DRUM SCANNER	*				
(5)	371	371DN	DRUM COUNTER			*	*	
(1)	664	664CrN1	*OLD* CRATE COUNTER	*		*		
(3)	664	664CrN2	*NEW* CRATE COUNTER			*	*	
(1)	707	707DS	SEGMENTED DRUM SCANNER	*				
(3)	707	707CN	COINCIDENCE COUNTER			*		
(1)	771	771CS	CAN SCAN I	*				
(1)	771	771DS	SEGMENTED DRUM SCANNER	*				
(1)	771	771CA1	CAN COUNTER		*			
(1)	771	771CA2	HELIX COUNTER		*			
(1)	771	771DA	SOUTH DRUM COUNTER		*			
(4)	771	771DN	NORTH DRUM COUNTER			*	*	
(1)	776	776CS	CAN SCAN II	*				
(4)	776	776DA	*LOSAC*		*			

NEW NAME: EXAMPLE: 664CrN1

- Counter number for that type counter in building.
- Type assay: S=segmented gamma scan; A=low resolution gamma assay; N=neutron method
- Type container: C=can; D=drum; Cr=crate
- Building number for detector system

'KEY'

1-Existing and operational	4-New system. Ordered but not installed (past the planning stage)
2-Old system. Not yet operational	5-New system. Planned
3-New system. Not yet operational	

060	oxide	374	blacktop, concrete, dirt, sand	432	resin, cemented
062	oxide heel	375	oil, dry	440	glass
067	oxide in small stacker can	376	cemented insulation	441	rashing rings
290	filter sludge	377	coarse fire brick	442	rashing rings, leached
292	cemented sludge	378	pulverized fire brick	480	light non-SS metal (Al, Cu, SS, Fe)
299	miscellaneous sludge	390	unpulverized slag	481	light non-SS metal, leached
300	graphite molds	391	unpulverized slag and crucible	484	scrap metal, classified shapes non-SS
301	graphite cores	392	unpulverized sand, slag and crucible	486	classified tooling for disposal
302	benelex and plexiglass	393	sand, slag and crucible heel	490	hepa filters
310	graphite, pulverized or fines	394	sand from button breakout (371)		
311	graphite heels	395	unpulverized slag and crucible		
312	graphite, coarse	396	pulverized slag		
320	heavy, non-SS metal (Ta, W, Pb, Pt)	397	pulverized slag and crucible		
328	ful-flo filters (from incinerator)	398	pulverized slag and crucible		
330	dry combustibles	399	pulverized slag and crucible		
331	ful-flo filters (not from incinerator)	404	molten salt, Ca, Zn, K		
332	oily sludge	405	molten salt, unknown and unpulverized		
333	calcium metal	406	molten salt, unknown and pulverized		
334	fire blankets	407	molten salt 8% unpulverized		
335	filters, 8 x 8	408	molten salt 8% pulverized		
336	wet combustibles	409	molten salt 30% unpulverized		
337	plastic, washables, etc.	410	molten salt 30% pulverized		
338	insulation	411	electrorefined salt		
339	leaded dry box gloves	420	incinerator ash, virgin		
340	sludge from size reduction	421	ash heel		
369	leco heels	422	soot		
370	leco crucible	423	soot heel		
371	fire brick	425	fluid bed ash		
372	grit	430	ion column resin		
373	fire brick heels	431	resin, leached		